



# **ENERGY PROGRAM REPORT**

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**1 OCTOBER 1976 - 30 SEPTEMBER 1977**

**U.S. DEPARTMENT OF ENERGY**  
**Assistant Secretary for Energy Technology**  
**Office of Fossil Energy**

**AUGUST 1978**



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WASHINGTON, D.C. 20545**



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# *FOREWORD*

The mission of the Fossil Energy Program of the Department of Energy is to ensure the efficient development of processes for using our Nation's fossil fuel resources. We want to make available proven technologies that — when commercialized — will help assure a dependable supply of socially and environmentally acceptable energy from these resources.

The formation of the Department of Energy on October 1, 1977, consolidated Government fossil energy research and development efforts and located them in the Office of Fossil Energy, which is a part of Energy Technology. In addition to the programs formerly included under the Assistant Administrator for Fossil Energy in the Energy Research and Development Administration (ERDA), we have added programs involving: new mining and coal preparation techniques, formerly a part of the Bureau of Mines; research efforts to make coal more environmentally acceptable, previously conducted by the Environmental Protection Agency; and utilization programs involving fuel cells, heat engines, and cogeneration, formerly under the Assistant Administrator for Conservation in ERDA. This volume describes the FY 1977 activities of all the programs that are now a part of Fossil Energy.

Since FY 1977 the focus of the Fossil Energy Program has changed. Our major objective now and for the foreseeable future is to develop alternative supply technologies that can substitute for liquid hydrocarbon fuels to offset the effects of an anticipated petroleum shortage. This includes more acceptable ways of using coal in applications now using petroleum. Another major thrust is concerned with assuring an adequate supply of gaseous fuels. Here our efforts are in both new sources of supply and the production of gas from coal. We are also forcefully pursuing the development of programs to facilitate the increased direct use of coal. In addition to these substantive changes, project implementation and execution responsibilities are being decentralized, and Energy Technology Centers (ETCs) in the field will play an increasingly important role in program execution.

Although energy costs are expected to increase, an effective Fossil Energy Program can help provide dependable energy supplies while holding cost increases to a minimum. We can accomplish this both by developing technologies that use energy more efficiently and by developing new supply technologies that more fully utilize our remaining fossil energy resources. Thus, we will help minimize the impact of rising energy prices, reduce U.S. dependence on imported petroleum, and ease the transition to more abundant domestic energy sources.

*George Fumich, Jr.*  
*Program Director for Fossil Energy*

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## EDITOR'S NOTE

This report is an integral part of the documentation system of the Fossil Energy Program of the Department of Energy. It contains descriptions of each contract and project, arranged in conformance with planning and budgetary documents. A special section is devoted to University contracts that are part of the Advanced Research and Supporting Technology Program. The section is arranged alphabetically by state. Other University contracts that are vital parts of specific programs appear in the description of that program (e.g., under Liquefaction). Many of the contracts and projects described herein were included in last year's version of this report, ERDA 77-70. Readers interested in more information on program structure and planning are referred to DOE/ET-0013, *Fossil Energy Research and Development Program of the U.S. Department of Energy*. A list of acronyms and abbreviations (pp. 623-628), a glossary of terms (pp. 629-632), and an index of companies and institutions (pp. 633-635), complete this report.

Projects being conducted by Energy Research Centers or National Laboratories are described at least quarterly in reports issued by these organizations. The results of contracts are reported at various intervals, depending on the type of contract, but at least annually. These reports are not listed individually in the "Publications" sections but are available from the National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia 22161. The Department of Energy also publishes several abstract journals:

- *Fossil Energy Update* (monthly, National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia 22161)
- *Energy Research Abstracts* (biweekly, Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402)
- *Energy Abstracts for Policy Analysis* (monthly, Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402). "

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*H. Neal Dunning*  
*Field Coordination, Fossil Energy*

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# ***COAL MINING AND PREPARATION***

These activities in coal mining and preparation were added to the Fossil Energy Program late in 1977 from the Department of the Interior, Bureau of Mines; therefore, specific contract and project reports will not be provided. Rather, general activities will be discussed to give a better overall understanding of the Fossil Energy Program that now involves technology on the total fuel cycle from mining through end use.

## ***UNDERGROUND COAL MINING***

Underground coal mining activities can be divided into the following four general areas: mine planning and development, production mining, transport, and other support. Coal mine planning impacts the range of decisions that must be made in deciding how a particular area of coal will be recovered. Coal mine development involves the cutting of the access openings necessary to allow a production mining system to be applied. These access openings provide for ventilation, men and materials transportation, and coal transportation. They may be vertical or horizontal shafts (in-mine development). In addition, the subject of methane drainage from the coal bed prior to mining is considered to be in the coal mine development area. The majority of underground coal production in the United States results from two production methods—room and pillar mining and panel mining. Room and pillar is the source of 95 percent of U.S. underground coal. Panel mining, the source of the remaining 5 percent, offers the potential for increased productivity, worker safety, and decreased environmental impacts; therefore, it is being given R&D focus in the program.

***COAL MINE SHAFT DEVELOPMENT (MINE PLANNING AND DEVELOPMENT)*** – A key element in underground mining is the preparatory excavation required to construct ventilation shafts, man and material access shafts, and in-mine roadways (entries) requisite to coal production. A program for the development of mechanized shaft-sinking systems to reduce the associated time and cost was initiated in 1975. This effort emphasizes development of: a downhole-powered (power supply at the cutting head) system for construction of man and material shafts, exceeding 20-ft diameter, commensurate with the needs of major new mines; a surface-powered system for construction of shafts up to 20-ft diameter (a practical upper limit for such systems) for ventilation needs and lesser man and material requirements. Both systems have the design capability to sink the shafts blind; that is, without having to depend on previous development in the mine itself.

The *Blind Shaft Borer* (BSB) is a system with the design capability of sinking greater than 20-ft-diameter shafts to depths of 2000 ft, the minimum required to accommodate projected production capacities and increased depth of reserves. Fabrication of the BSB and related support systems is in process. The major support systems are the secondary haulage (top of BSB to surface) and the lining systems (presently, 12-in.-thick concrete). Even using state-of-the-art support systems for the first field trials, shaft construction rates of 36 ft/d are projected, an order of magnitude improvement over current rates.

Fabrication has begun on a surface-powered *rotary drilling system* capable of excavating up to 20-ft-diameter ventilation shafts and remotely placing a 12-in.-thick concrete lining. This method uses scaled-up oil-well drilling technology where men stay safely on the surface during the drilling

operation. The lining placement system that is part of this development borrows from the civil construction and electronics industries, combining slip-forming and remote sensing and controls. The rate of excavation is expected to be comparable to that of the BSB.

**IN-MINE DEVELOPMENT (MINE PLANNING AND DEVELOPMENT)** – Development of new areas for coal production typically involves the use of a continuous miner advancing a heading in sets of five roadways (entries). Ten to fifteen entries are generally required on a main heading. The large number of entries that are driven is largely caused by ventilation and haulage demands and restrictions on entry dimensions. Continuous miners are not designed to cut roof or floor rocks; therefore, entry height is limited by coal seam thickness, and width is limited by roof stability. In addition, crosscuts between entries must be driven at intervals of 100 ft or less to meet current regulations. The large number of entries and crosscuts required, combined with roof support and ventilation considerations, dictates that the majority of time be spent in moving the continuous miner from one location to another and in preparing to cut coal. This is particularly true in development operations, because the coal pillars left between entries must be large to provide support for the life of the mine. The result is an extremely slow rate of development. Despite the relatively large pillars left, ground control problems persist because of the number of entries involved. Continuous maintenance is required, therefore, and disruption of service results. Research activities to alleviate the in-mine development problems have been done in part under production-related research; that is, the development of a better continuous miner and the integration of bolting. Meanwhile, in the development area other basic equipment, technology, and approaches have been examined. Evaluations to date have focused on the potential of three basic applications: slope access from the surface and main entry development (tunnel boring); submain and panel development involving both the cutting of coal and roof and/or floor strata (mixed face); and submain and panel development restricted to the coal seam (in-seam).

As a step toward alleviating development problems, a cost-sharing contract was undertaken to examine the technical and economic feasibility of applying *Tunnel Boring Machines* (TBMs) to mine development. TBMs came into their own in the early 1970s in the civil construction industry, finding wide use in the construction of transportation tunnels, sewer systems, and other underground operations involving large-diameter openings in both hard and soft strata. TBMs show potential for mine development because of the possible speed of excavation, often hundreds of ft/d in civil works projects; the stability of the circular opening; and the fact that large openings could be driven independent of strata considerations. As to the latter, it was determined that, from a ventilation standpoint, the 18-ft-diameter TBM chosen for the cost-sharing project provided the equivalent of five entries in the particular mine involved. Stability of opening is important from a maintenance (and safety) standpoint, and speed is essential in addressing the basic development problem. Under this joint effort, a 6000-ft-long tunnel is being bored off the East Mains of the Federal No. 2 Mine to connect existing mine workings with an air shaft and to open the area for longwall mining. Technical and economic feasibility is being evaluated in view of the constraints relative to the hazardous environment and requirements imposed by Federal and State mining laws. Another major objective is to obtain the data requisite to developing specifications for a TBM system optimally designed for the coal industry. The system has now operated safely for 2600 ft and remains in an operational state in a region previously shown to be too gassy for conventional development.



Where shorter distances are involved, as in submains (laterals) or panel development, a system more mobile and maneuverable than TBMs is required. In the lower seam-height range of 30 to 60 in., there is particular need for a system that can cut both the coal and adjacent strata to reduce the number of entries required and to provide greater freedom of movement. There has been an ongoing examination of equipment, technology, and concepts both foreign (primarily through agreements with England, West Germany, and Poland) and domestic having potential for this application. For example, investigations have been conducted on rock-cutting machines such as roadheaders, dentheaders, and high-energy impactors; augers as a crosscut excavation tool; monolithic packing systems, mechanical packers and foam concrete placement systems for roof support; self-advancing and manually set temporary roof supports; concepts employing combinations of coal and rock cutting equipment; latest developments in roof-bolting systems; and new technologies, such as water jets, to assist mechanical cutting of rock. Available technology has been explored, shortcomings have been noted, and efforts initiated to advance that technology where required. Emphasis, however, will not be placed on incorporating present technology into an efficient system meeting industry needs.

Two basic approaches will be pursued in the system development; a *Single-Face Multiple-Entry* (SFME) concept; and a *Multiple-Face Continuous-Drivage* (MFCD) concept. In the SFME approach, a wide face is cut by a single cutting machine, and a number of entries are created to the rear by artificially placed structural walls. These walls provide the alternate escapeways and ventilation splits required by mining law. The MFCD approach is to achieve a continuous advance of more than one cutting machine and entry with a coal pillar or pillars between. This will require bringing to bear sensing and control technology to safely operate more than one cutting machine on a split of air, auxiliary cutting machines for crosscuts so the primary machines are not disrupted, and temporary support systems to reduce the interdependency of cutting and roof bolting.

Where seam thickness permits efficient shaft development in the coal, the same basic approaches as outlined above will be pursued using coal-cutting machines. Developments from production-related research, such as the miner/bolters, may be incorporated into the systems, or promising new machines, such as the *inseam miner* developed in England, may be used.

**METHANE DRAINAGE** – Tremendous quantities of methane within coal seams and adjacent strata represent a vast energy resource, but also pose a major safety hazard in coal mining. The recent Scotia and earlier Farmington mine disasters serve as constant reminders of the dangers related to methane liberation in coal mines. Most of the extensive precautions taken regarding ventilation, haulage, and mining methods are in response to this hazard. To keep mines at safe methane concentration levels, circulation of large amounts of air throughout the mine is required. Regulations require the shutdown of equipment when methane concentrations reach 1 percent. Even with seemingly adequate air flow rates in production sections, cutting is often slowed or halted because of methane liberation, resulting in reduced efficiency and increased costs. Degasification, removing most of the methane from permeable coal before mining, can alleviate methane-related mining problems and also tap a significant natural resource. In a program begun several years ago, three basic degasification techniques are being developed: vertical wells; directional drilling; and horizontal drilling.

The *vertical wells method* involves the drilling of small-bore vertical wells spaced throughout a mine property and hydraulically stimulated to propagate flow paths for the methane gas. In the



Pittsburgh and Mary Lee coal beds, vertical wells have been effective in removing large quantities of gas, to the point where commercialization of the product appears economically feasible. This method cannot totally bleed off the methane in a region within a reasonable period of time. It can, however, isolate a region from the methane flow path by offering a path of less resistance (up the vertical wells). Efforts will continue to measure the effectiveness of the basic method in other coal seams and to determine optimum well spacing.

The *directional drilling method*, an extension of the vertical well technique, involves the drilling of a number of small-bore wells from a single location, thus reducing time and cost for site preparation, mobilization, and securing property rights for well location. In the rough terrain of the eastern coal region, this can be a considerable savings. Directional drilling methods were developed for oil fields. Although the technology exists, the effectiveness of the borehole, the stimulation methods, and dewatering techniques must be determined so the required pattern can be established and expected economies assessed.

The *horizontal drilling method* involves the drilling of long horizontal small-bore wells from the bottom of a large diameter shaft. Horizontal drilling from a shaft bottom is known to be effective. Because a large shaft is required, the technique is only economical when carried out in conjunction with mine development. At Federal No. 2 Mine, a series of horizontal boreholes, draining into a wide, multipurpose vertical borehole and then into an air shaft, have been effective in producing large quantities of methane for several years.

**ROOM AND PILLAR (PRODUCTION MINING)**— The initial production stage of a room and pillar operation involves the driving of a set of parallel interconnected entries (tunnels). Mining is accomplished by the cyclic performance of operations in cutting, loading, and roof support. The interconnection of the entries is necessitated by the requirement for closed loops for ventilation and produces a checkerboard pattern of pillars. The second production stage of the room and pillar mining method involves partial mining of the remaining pillars of coal. The continuous miners used in room and pillar mining are capable of producing 6 to 12 t/min.; however, they typically average only half that production for only 20 percent of the shift because of the nature of the method as currently practiced. The thrust of the room and pillar effort is to develop systems that will permit more-truly-continuous mining throughout the shift. The initial goal is to reduce lost production time through increased system reliability and improved combinations/cycling of machine functions. Once this is accomplished, emphasis will shift to raising the average production rate closer to the potential of the basic machinery.

A *continuous roof supports* effort, an integrated part of room and pillar mining, deals with better permanent and temporary roof support methods. A total system approach is used to get first-time prototype machines to the field quickly. These systems use continuous miners to which a simultaneous roof bolting capability has been added (miner/bolters). Laboratory testing currently is underway. Meanwhile, major improvements are being made in the components used on these new complex systems. The component development work includes contracts for longer-than-seam-height drills, high-speed water jet drills, and bolter modules. Each of these elements is being perfected through a series of laboratory and underground tests by which design and reliability problems can be isolated and solved. This parallel effort allows a methodical development plan for the components simultaneously with investigation of system problems on the miner/bolters. As the compo-

nents reach the high reliability needed for new complex systems, they will be combined to form a new advanced generation of miner/bolters.

A *remote-controlled continuous miner* effort is progressing on a development plan similar to the roof support effort. The project, which includes both system and component development contracts, is aimed at the elimination of decision-making constraints in the operation of the miner. The Automated Extraction System (AES) is a full-face programmable continuous miner with roof bolting capability and self-advancing ventilation. In the component development work, studies are being done to determine the degree of automation that can be implemented in underground systems and to develop scenarios for these systems. Some of the ancillary items being developed include coal interface detectors, guidance systems, and maintenance management systems.

Nearly 75 percent of underground room and pillar mines in the U.S. use shuttle cars to transport mined coal from the working face to a secondary haulage system. The majority of these units are electric powered and, to transmit power to the vehicle, use a trailing cable from the reel on the shuttle car to a stationary tie-off point. This umbilical cord connection is a safety hazard, restricts underground travel, and limits the system to two operating vehicles. The introduction of continuous mining machines prompted the mining industry to reassess shuttle-car batch-type haulage. Between location changes, the continuous miner can cut and load coal on a nearly continuous basis; however, after loading a shuttle car, the high-capacity machine must wait while the second shuttle car is spotted under the miner tailboom. Delays in the haulage system, usually resulting from waiting for shuttle cars, take 25 percent of the continuous miner cycle-time. Computer simulations of a face operation indicate that up to a 45 percent increase in coal production could be realized through the use of a *continuous haulage system* associated with a secondary haulage system to handle the increased load. Several types of continuous haulage systems are under study and development. They include a floor-mounted automated bridge conveyor train, monorail-mounted bridge conveyor system, extensible-belt bridge conveyors, flexible-belt conveyor train, coal injectors for hydraulic pipeline transport of coal from the mine face, and a multiple unit continuous haulage system.

**PANEL (PRODUCTION MINING)** – Panel mining is a high-production approach to coal mining that is widely used in Europe but has found only recent acceptance in the United States. To date, this method accounts for only 4 percent of the coal mined by underground methods. Basically, large rectangular blocks of coal are defined and extracted by successively slicing one of the sides. For example, in retreat longwall panel mining, the predominant method now used in the United States, the complete block of coal to be mined is usually 300- to 500-ft wide and 3000- to 6000-ft long. Up to 4000 t/shift have been extracted from a 7-ft seam. Typically, however, only 700 to 900 t/shift are realized because of equipment problems and disruptive geological conditions. High capital requirement for the equipment is another constraint to the use of longwall mining. The panel mining effort includes three major projects: improving longwall system performance; decreasing longwall system costs; and technology demonstrations to lower the perceived risk of panel mining.

Current *longwall mining* practice in this country involves the cutting of a thin slice of coal, generally less than 30 in., from a block of coal about 500-ft long. One objective of this project is to realize a 30 to 100 percent increase in the thickness of this slice for the majority of the longwall faces in operation. A study is underway to establish the optimum value and upper limit of operation

for the slice thickness in longwall mining operations. Another way to improve system performance is to increase the total length of face sliced per shift. Current longwall mining systems in the United States cut approximately 2500 ft of face on the average day that the mining system is in a production mode. The second objective of this project is a 100 percent increase in this length on the longwall faces (about 25 percent) geologically compatible for such increases.

The capital cost of a longwall system can be as much as five times the cost of a single continuous miner production unit. Of this longwall capital equipment cost, 80 percent can be attributed to the roof support system. The objective of this project is to reduce roof support costs for the newly introduced longwall faces. A study is being done to determine if roof supports are significantly underutilized and to establish more precise methods of estimating support requirements.

**NOVEL SYSTEMS (PRODUCTION MINING)** – Additional attention is focused on the modification and application of existing mining systems to unique mining situations; that is, thick, multiple, and steeply pitching seams, and development of advanced mining systems that offer economic advantages over current systems. Since the vast majority of coal mined underground is produced from coal seams less than 10-ft thick, the coal industry techniques and equipment that have been developed are better suited to these thinner seams. In mining thick seam coal (10- to 40-ft) with present techniques, there has to be a compromise in recovery, safety, or profit; therefore, good recovery is usually sacrificed. Recovery percentages below 30 percent might be expected when present room and pillar techniques are applied to a coal seam 20-ft thick or more; the remaining 70 percent or more of the coal in place is lost. The effort to find a better way to mine thick coal seams is important from the standpoint of increased production of coal and improved resource recovery.

It is projected that site selection will be completed and a detailed mine design plan finalized for the first U.S. application tests of the longwall multilift method for extracting thick underground coal in 1979. The method consists of working a panel of longwall mining face starting at the top of the thick coal deposit and working down in horizontal slices. For example, in a 20-ft seam the first pass extracts the upper half of the seam and lays a screen mesh down on top of that remaining. This screen acts as an artificial roof when the bottom half of the seam is mined. Resource recovery using the longwall multilift method will approach 60 percent, twice that of conventional room and pillar methods. Efforts will continue to develop methods to mine the large amounts of coal in steeply pitching seams, previously unrecoverable because the steep operating angle ( $> 25^{\circ}$ ) precludes use of continuous mining methods. The alternative of longwall mining will be tested in Colorado.

Development and testing of a prototype longwall sublevel caving technique will continue. A field test will be the first application of such a system in the United States. The method consists of working a longwall face along the bottom of the thick seam and drawing or recovering the top coal from the rubble as it caves into the extracted cut (gob). Gobbed coal is recovered on a conveyor either by gravitational flow or mechanically stimulated methods. Projected resource recovery for this method will approach 80 to 85 percent.

*The Sublevel Caving with Pillar Extraction (SCPE)* method is a modification of pillar mining, with the addition of top coal recovery. Mine openings will be driven against the bottom of the seam

using room and pillar techniques. Top coal recovery is accomplished on retreat by drilling and blasting the top coal in increments and loading out the fallen top coal with a continuous miner.

Efforts in high-volume hydraulic mining address the problems of recovering coal that lies in moderate to thick seams ( $\geq 6$  ft) having pitch angles greater than 4 degrees. Most technology for hydraulic mining has been used in other countries. This effort will explore the technique's applicability to U.S. conditions. Studies have indicated that this method may be economically advantageous over other forms of mining for pitching coal seam conditions. Development work is underway on high-energy low-volume hydraulic jet augmentation of mechanical mining systems. Use of cavitating, percussive, and continuous water jets is being investigated. An effort is underway to develop the best combination of a low-volume jetting system with a boom-type miner to determine if the jet increases production in coal and rock.

**TRANSPORT AND OTHER SUPPORT** – Improved haulage systems to effectively move men, supplies, and mined coal between the mine working section and the surface are of utmost importance in overall development of a cost-effective underground coal mining system. The major project being studied is the *automated rail haulage system*. This system uses unit trains that circulate within the mine, without operator assistance, from various loading sites to the unloading point and back to the same or alternative site. The system requires a locomotive designed specifically for automation, with controls and sensors in the entire system necessary for unmanned operation. The overall system eliminates workers from the most hazardous portions of the rail haulage operation. The automated rail haulage system is expected to reduce rail haulage costs 7 to 12 percent, to increase overall in-mine productivity 3 to 6.5 percent, and to reduce haulage accidents by 50 percent.

Surface testing of new and modified underground coal mining equipment has been limited, in most cases, to functional checkout of major systems with no load applied. As a result, the first real tests of equipment performance under load are now conducted in underground production operations. A *Surface Test Facility* under construction at Bruceton will provide capability to conduct and evaluate efforts above ground on mine equipment and systems in a simulated underground mining environment, which will expedite the introduction of safer and more efficient technology in the underground mining operation. Planned tasks for the facility include evaluation of the longwall cutter/loader for pitch, roll, yaw, and haulage parameters; evaluation of continuous miner for stability of controls and for reliability and effectiveness of the interface detectors and guidance control components; and study of the variable wall miner to determine its cutting and conveying capability.

## ***SURFACE COAL MINING***

The Surface Coal Mining projects are divided into two areas: area mining and contour mining.

**AREA MINING** – Area mining involves making a trench or box cut through the overburden to expose the coal deposit, which is then removed. The first cut may be extended to the limits of the property or the deposit. As each succeeding parallel cut is made, the spoil (overburden) is deposited in the cut just excavated. The final cut leaves an open trench as deep as the thickness of the overburden plus the coal recovered, bounded on one side by the last spoil bank and on the other by

the undisturbed highwall; therefore, area mining, unless graded or leveled, usually resembles the ridges of a gigantic washboard. Historically, it has been practiced on relatively flat terrain. A major thrust of this subactivity is to investigate application of area mining techniques to thick, multiple, and steeply pitching seams of coal. Projects being pursued include mine design and planning, thick- and multiple-seam mining, and conventional mining equipment improvements.

The *mine design and planning* project emphasizes the more effective use of available mining and reclamation equipment and technology, achieved largely through improvements in equipment combinations and design of layout and haulage configurations, and through mine site evaluation and preplanning of mining and reclamation activities. Area mining requirements for moderately and steeply pitching coal seams are being analyzed. Design parameters outlining site selection criteria will be assembled to combine proper equipment and mining methods for efficiently extracting surface coal reserves from areas where both the coal seam and surface topography exceed a 9-degree pitch. A complementary effort to analyze the production efficiency of terrace-pit mining systems is also underway. Terrace-pit mining is a method for single or multiple coal seams where terrace or bench levels are used in the open-pit design to optimize mine equipment combinations. It is advantageous because spoil placement is easily controlled, grading can be concurrent with production, equipment delivery time is shorter than with dragline methods, production loss from downtime is less, and spoil rehandling problems are eliminated.

A project in *thick- and multiple-seam* mining is oriented primarily toward exploiting surface coal reserves west of the Mississippi. Even though geographic and geologic conditions are diverse in these new western surface coal mines, two basic mining methods—dragline casting and shovel and truck—have historically been used. Draglines with booms of up to 300 ft and buckets with 60 to 70 cubic yard capacities are used. While draglines of this size are capable of digging overburden depths of 150 to 200 ft in one lift, they cannot effectively dispose of the spoil without rehandling. Truck-and-shovel operations provide a more flexible mining system than dragline casting, but overburden handling can be three to four times more costly. The thick- and multiple-seam mining project is developing three new mining equipment systems designed to increase productivity at competitive economic cost: mobile overburden conveyors and continuous spreaders in conjunction with existing excavators; a continuous high-volume overburden excavation and handling system; and a continuous high-volume coal excavation and handling system. The entire mining cycle from ground preparation and excavation through the transport, dumping, and leveling phases is considered and integrated in developing equipment specifications for each of these systems. Four competing mobile cross-pit conveying systems have been designed that use a hopper to handle material from a dragline and a spreader to distribute the spoil. These four systems are: an inclined bridge with a shuttle conveyor for spreading spoil; a direct bridge from the highwall to the spoil bank; an inclined bridge with spreader; and a steep-angle conveyor using an overlapping belt.

Although a CPCS is a more efficient and economical spoil removal system than shovel/truck or conventional dragline methods, it is still costly since it requires a relatively expensive supplementary hopper to convert the system from a cyclic to a continuous operation. A truly continuous mining system could be even more efficient, eliminating the need for a dual material handling capacity and the cyclic excavating process. Therefore, a variety of highly mechanized equipment that can continuously excavate large volumes of overburden is being developed for use in the west. Depending upon the nature of the primary machine configuration involved, these continuous mining machines can be classified into four categories: boom-type bucket-wheel excavators; integral wheel bucket-

wheel excavators; drum shearer and auger-cutter type excavators; and vertical-arm belt-loading excavators. Several of these machines can potentially be developed for use in both coal and overburden extraction. The project is pursuing development of two distinct systems, a continuous equipment system for coal and a continuous equipment system for overburden. Machines selected for use in either coal or overburden removal will be examined to determine their application to both systems. Feasibility analyses and component testing of different concepts will be carried out during FY 1979. Based upon the results of these activities, detailed designs will be developed and the selected machines will be fabricated.

The *conventional mining equipment improvements* project stresses modification of equipment components, development of better maintenance techniques, upgrading of equipment operators' skills, and development of control assist devices for equipment operators. These efforts are geared to reduce operating costs and ultimately make existing surface coal mines more competitive with other energy sources. A wire rope fatigue testing machine has been developed that will be used to test dragline wire rope life during FY 1978. The machine is capable of testing 3-in.-diameter ropes with loads of up to 500,000 lb. Application of these test results should reduce industry's wire rope maintenance costs, which are estimated to be \$15 million per year. Efforts are also under way in dragline boom fatigue, dragline lubrication, and optimal dragline operating techniques. Based on the results of these studies, equipment will be designed and field tests will be initiated in FY 1980. The dozer draft power sensor, an electronic device attached to a tractor dozer, has been developed to measure the velocity of the tractor and the load on the dozer blade. It uses these factors to calculate a work rate (horsepower output). By using this device, an operator can maintain maximum production rates indefinitely. Initial field tests have been encouraging, and extended field tests will be conducted.

**CONTOUR MINING** – Contour mining consists of removing the overburden above the coal bed by starting at the coal outcrop and proceeding along the hillside. After the deposit is exposed and removed by this first cut, additional cuts are made until the ratio of overburden to coal brings the operation to a halt. This type of mining creates a shelf or "bench" on the hillside. On the inside, it is bordered by the highwall, which may range from a few to more than 100 ft in height; on the opposite or outer side, it is bordered by a rim below which there is frequently a precipitous downslope that has been covered by spoil material cast down the hillside. Projects currently under way include contour mining conveyor systems, crossridge demonstration, and highwall mining systems.

The *contour mining conveyor systems* project involves development of two separate systems: a low wall conveyor haulage system and a mountaintop conveyor system. The objective of developing these systems is to promote continuous overburden and coal removal activities at surface mines operating in steep terrain. These systems can potentially be cost effective while achieving excellent reclamation and environmental improvements. A low wall conveyor haulage system has been developed for the recovery of coal in steep-slope areas of Appalachia. This method facilitates return of the mined area to its approximate original contour while minimizing or eliminating acid spoil problems. A series of field tests of this system will be carried out during FY 1978 and FY 1979. The system's range of operations will be investigated in FY 1978; variations for application in other areas will be designed in FY 1979. The mountaintop conveyor system will enhance resource recovery while achieving excellent environmental and reclamation standards at surface mines

operating in steep terrain. Systems and equipment have been analyzed and their potential for practical application determined.

A project in *crossridge mountaintop demonstration* involves a surface mining technique for mountaintop coal where the mining direction is perpendicular to the long axis of the ridge line. Compared to conventional mountaintop removal, the crossridge technique increases resource recovery, reduces operating costs, reduces the area disturbed by mining, and facilitates compliance with reclamation requirements. A demonstration using existing equipment will be carried out, in cooperation with a coal company, to verify the feasibility of this technique.

The *highwall mining system* project will employ an extended depth auger to exploit the large quantities of coal that are left behind the highwall with conventional mining methods. Development of an extended depth auger will increase the total recovery of coal beds augered, provide favorable economics for total reclamation, increase coal production, and increase economically minable coal reserves. The auger being considered will be able to remotely mine coal in excess of 500 ft from the highwall. Efforts are concentrated on developing sophisticated guidance systems for coal-rock boundary sensing and cut-thickness determination. Preliminary designs have been completed for two competing concepts, and a model of one machine has been constructed.

**RECLAMATION AND SUPPORTING STUDIES** – A number of studies will be undertaken to support implementation of the more restrictive reclamation requirements of the Surface Mining Control and Reclamation Act of 1977 (PL 95-87). One group of studies will develop basic information regarding extent and magnitude of the environmental impact of surface mining. Well-documented information from scientific studies will permit determinations of whether changes in regulations are needed. Typical studies include hydrology and water quality of watersheds subjected to surface mining, and effects of noise and vibration from blasting. A second group of studies will provide mine operators with information regarding technical and economically efficient methods of complying with various aspects of reclamation standards. Typical projects address the establishment of stable long-term vegetative cover, blasting techniques to minimize vibrations and noise, and the quantity of topsoil necessary for effective reclamation.

## **COAL PREPARATION**

The Clean Air Act of 1970 clearly mandated research in coal preparation. This research focuses on meeting SO<sub>x</sub> emission standards; identifying and monitoring trace fugitive elements present in the raw coal (i.e., coal preparation refuse, clean coal, fly ash, or emission exhaust); and meeting stringent standards on environmental effects of the coal preparation process itself. Four projects have been established: process test facility, coal preparation technology, waste utilization and control, and supporting studies.



depending on the flow scheme used. Process flexibility is a prime design requisite of this plant, which will use off-the-shelf commercial equipment. New coal-washing circuits and crushing, screening, dewatering, and cleaning equipment will be evaluated. The plant will be used to process coal from various regions and to provide the optimum operating conditions necessary to produce a coal of acceptable sulfur, ash, and trace element content. An additional function of the demonstration plant will be to produce ton lots of specification coal for combustion testing, stack gas scrubbing work, or as feedstock to coal conversion processes.

**COAL PREPARATION TECHNOLOGY** — Improved and innovative physical separation technologies are being developed to separate fine coal from pyrite effectively. Desulfurization can be accomplished through physical processes such as gravity separation flotation, and magnetic separation techniques or through varying chemical techniques that will remove most of the pyrite sulfur plus some of the organic sulfur intimately bonded within the coal molecule. With the ever-increasing growth in production of fine-size coal as a result of mining techniques or simply as a means to liberate impurities, it has become necessary to put the fine material in a more convenient form for handling, transporting, and storing. Preparation requirements for lignite and subbituminous coals include sodium reduction as well as the dewatering and pelletizing or briquetting necessary for bituminous coal. Efforts to reconstitute bituminous coal will also be pursued, focusing on briquetting and pelletizing. Since considerable knowledge has been accumulated concerning anthracite briquetting and lignite pelletizing, these efforts will concentrate on the engineering improvements of a known technology. Additionally, initial transport feasibility studies associated with coal/gelled oil mixtures and other fine coal transport systems will be initiated.

**WASTE UTILIZATION AND CONTROL** — Surface ground waters are contaminated from coal by materials leached from waste piles; pollutants in the form of suspended solids from preparation plants; and materials leached from surface water percolating through ground contaminated with airborne fallout from stack gases. Studies will be initiated to develop a low-cost method for dewatering fine wastes to obviate the need for tailing ponds. These studies will concentrate on the optimization of a filter design and disposal system, plus the pelletizing and firing of the fine waste in a fluidized bed to utilize the heat associated with waste. Pollution of land downstream from stack plumes will be minimized as efforts continue to reduce the hazardous trace elements in coal by precombustion cleaning.

**SUPPORTING STUDIES** — Studies will be continued to identify and characterize coal minerals, carbon/sulfur compounds, and trace elements found in various coal seams in the United States. Washability studies, which show the release or liberation of pyrite at various screen sizes, will be continued until samples from most of the major seams have been tested.





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# LIQUEFACTION

Processes for converting various types of coal to synthetic liquid fuels are steadily being improved so that clean fuels can be produced commercially when required. Emphasis is on the production of fuels suitable for firing industrial and electric utility boilers and on synthetic crude oils for upgrading to liquids for use in transportation and home heating. In addition, promising new processes are supported in laboratory research from which the most successful may be selected for development at larger scale.

Three processes have previously been selected and are now in the pilot plant phase. Collectively these three approaches are known as second-generation coal liquefaction processes, a category that includes Solvent Refined Coal (SRC), H-Coal, and Donor Solvent Processes. DOE is supporting operation of the (50-t/d coal) SRC pilot plant located at Fort Lewis (Tacoma), Washington, where the operating contractor is the Pittsburg & Midway Coal Mining Company. A smaller SRC plant is operated at Wilsonville, Alabama, by Southern Company Services, Inc., with support of DOE and the Electric Power Research Institute.

DOE and a consortium of industrial sponsors are supporting the design, construction, and operation of a 600-t/d (coal) H-Coal pilot plant at Catlettsburg, Kentucky. Responsibility for design of the plant is assigned to Hydrocarbon Research, Inc. Ashland Synthetic Fuels, Inc. will operate the plant.

Under a cooperative agreement, DOE and Exxon Research and Engineering Company together with other industrial partners are supporting development of the Donor Solvent Process. A.G. McKee Company is the engineering design and procurement contractor and Daniel Construction is the erection contractor for the 250-t/d (coal) pilot plant now under construction at Baytown, Texas. Carter Oil Corporation will operate the pilot plant.

The Cresap Test Facility in Cresap, West Virginia, has been placed in operation to evaluate liquefaction equipment and unit operations common to a number of coal liquefaction processes. The operating contractor is Liquefied Coal Development Corporation, a subsidiary of Fluor Engineers and Constructors, Inc.

Third-generation processes provide technical backup and significant potential improvements over second-generation processes. The Rocketdyne Division of Rockwell International is developing the technology for Partial Liquefaction by Direct Hydrogenation (Flash Liquefaction) in an entrained flow reactor and Occidental Research Corporation has evaluated the commercial potential of its Flash Pyrolysis Process.

A severe hydrocracking process using molten zinc chloride catalyst to produce naphtha range product is under development by Conoco Coal Development Company with the participation of Shell Development Company. The Lummus Company has investigated the use of a multistage ebullated-bed catalytic reactor in their Clean Industrial and Transportation Fuels from Coal Process, and U.S. Steel engineers and consultants have completed their development efforts with the Clean Coke Process. Also, the University of North Dakota is completing their study of the Solvent Refined Lignite process in a 0.6-t/d unit. Development work on the Synthoil process was completed

*HRI Process Development Unit Reactor Tower*



at the Pittsburgh Energy Research Center (PERC) by investigating the thermal reactions involved. The 10-t/d process development unit at PERC is nearing completion after which it will be available for further development of a promising third-generation process.

Development studies on the fluid-bed process for selective conversion of methanol to gasoline have been continued by Mobil Research and Development Corporation. Research projects involving process development efforts are underway at PERC where an important topic is an investigation of expandable catalysts. At the Grand Forks Energy Research Center, the effort is concentrated on approaches to the liquefaction of lignite.

Research to improve solid liquids separation using filtration techniques is conducted by Johns-Manville Sales Corporation, while projects to evaluate commercial processes for the fluid coking and gasification of high-ash residues of liquefaction are underway with the Exxon Research and Engineering Company and Texaco Development Corporation, respectively.

The Cities Service Research and Development Company is studying the feasibility of using a commercial-scale hydroprocessing unit to produce liquid fuels or refinery feedstock from a heavy solvent refined coal extract. UOP, Inc., is evaluating standard refinery techniques for the processing of coal-derived naphtha produced by the H-Coal and Donor Solvent Processes. Dow Chemical Company completed studies to evaluate the production of chemicals from coal using liquids from four coal liquefaction processes (COED, Synthoil, H-Coal, and SRC) via standard petrochemical techniques.

A number of advanced research projects are supported to investigate the role of catalysts, development of new catalysts, liquefaction mechanisms, conversion of coal gases to clean liquids, and processing of heavy residues.

#### PHASE I/II H-COAL INTEGRATED PILOT PLANT

HYDROCARBON RESEARCH, INC.

DOE - \$38,924,604; Participants\* - \$7,248,750

12/1/73 - 12/31/78

**OBJECTIVES** – This total program is designed to demonstrate the H-Coal process by constructing and operating an integrated H-Coal Pilot Plant at Catlettsburg, Kentucky. The facility will process up to 600 t/d of coal to produce either a synthetic crude oil or a boiler fuel oil. Specific objectives of the Phase I program are to design the pilot plant, purchase equipment, and obtain experimental verification of certain key design features at the HRI R&D center in Lawrenceville, N.J. Purchase of pilot plant equipment will be a Phase II activity.

**RECENT WORK AND ACCOMPLISHMENTS** – The final report on the completed experimental work was written, reviewed, and issued. Equipment purchase commitments totaled \$27.3 million by the end of the year. Engineering work covered by this contract was completed.

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\*Phase I funding participants are Ashland Oil, Inc., Atlantic Richfield Oil Co., The Commonwealth of Kentucky, Electric Power Research Institute, Shell Oil Co., Standard Oil Co., and Sun Oil Co.

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**PLANS FOR THE COMING YEAR** – The conclusion of the purchasing effort, amounting to approximately \$4 million, is the only remaining activity under this contract. Construction, field engineering and additional laboratory work are being performed under separate contracts, which are covered in the next several writeups.

## **PHASE II LABORATORY SUPPORT FOR H-COAL PROJECT**

**HYDROCARBON RESEARCH, INC.**  
DOE - \$5,658,145; Participants\* - \$1,053,689  
12/01/76 - 12/01/78

**OBJECTIVES** – The Phase II H-Coal laboratory work in 1977 was performed to provide maximum assurance of successful operation of the pilot plant under construction at Catlettsburg, Kentucky. Since enough coal is available to supply our energy needs for several centuries, developing H-Coal as a commercially viable process will significantly reduce dependence on other, uncertain energy sources; improve our balance of payments; and contribute to the economy by increasing the use of domestic coal resources.

**RECENT WORK AND ACCOMPLISHMENTS** – HRI began Phase II by modifying the Process Development Unit (PDU), upgrading facilities to increase personnel safety, and constructing a catalyst addition-withdrawal system. Simultaneously, a full-sized coldflow model of the pilot plant reactor was built that verified operability of improved reactor intervals at the high gas velocities expected in the 600-t/d pilot plant. Extensive changes to bench-scale units were initiated to upgrade their ability to support the overall program objectives. These modifications will enable use of these bench units to identify and evaluate catalysts that have characteristics making them suitable for use in the pilot plant. PDU Run 5 was the first operation in this contract period. It successfully demonstrated the ability to add and withdraw catalyst while in full operation to achieve equilibrium catalyst and reaction conditions. Process conditions and operating objectives simulated those that will be used in the pilot plant when it processes Illinois No. 6 coal in the syncrude mode. Samples were produced for a product analysis program at Mobil, and satisfactory agreement with previous correlation was established.

**PLANS FOR THE COMING YEAR** – When the bench-unit modifications are completed, in-depth catalyst evaluation studies will be started to evaluate improved catalysts for use in the pilot plant. PDU operations will be extended to test the operating modes and coals that will be used in the pilot plant, develop emergency operating procedures, and continue training pilot plant operating personnel. Engineering studies that started in 1977 will be completed.

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\*Phase II funding participants are Ashland Synthetic Fuels, Inc., Commonwealth of Kentucky, Conoco Coal Development Co., Electric Power Research Institute, Mobil Research and Development, and Standard Oil Co. of Indiana.

## H-COAL PILOT PLANT: PHASES II/III, CONSTRUCTION AND OPERATION

ASHLAND SYNTHETIC FUELS, INC.  
DOE - \$134,100,000; Participants\* - \$30,000,000\*\*  
8/16/76 - 12/31/82

**OBJECTIVES** — Construction of a commercial H-Coal plant having maximum reliability of operation with minimum capital and operating costs is the goal of Phase II of this program. Specific objectives are to construct a pilot plant with the design flexibility to process between 200 and 600 t/d of coal (depending on the mode of operation and the desired product) and then produce a maximum of 1800 bbl/d of coal-derived liquids. Phase III objectives are to demonstrate operability in both the syncrude and fuel oil modes of operation, establish yield correlations as a function of coal type and operating conditions, obtain data for each of the unit operations as required for scaleup to commercial size, and obtain samples and correlate data derived from the pilot plant. Records will be kept on equipment to facilitate comparison with design performance, suggest design changes, and evaluate maintenance requirements. Sufficient engineering will be provided to support an independent commercialization study of the H-Coal process. Further objectives include determining the metallurgical requirements of the process, evaluating the Lummus antisolvent deashing system and, potentially, other solid/liquid separation systems, improving design, defining plant maintenance requirements, determining environmental effects of the process, and evaluating the technical and economic feasibility of producing fuel oil, synthetic crude oil, and intermediate coal liquids by the H-Coal process.

The potential value of this process lies in three areas: high-sulfur coals, which cannot be used in conventional methods because of environmental considerations, are converted into useful low-sulfur coal-derived liquids; a large range of products are available by varying operational modes; and its ability to use different types of coal at different modes of operation, i.e., operational flexibility.

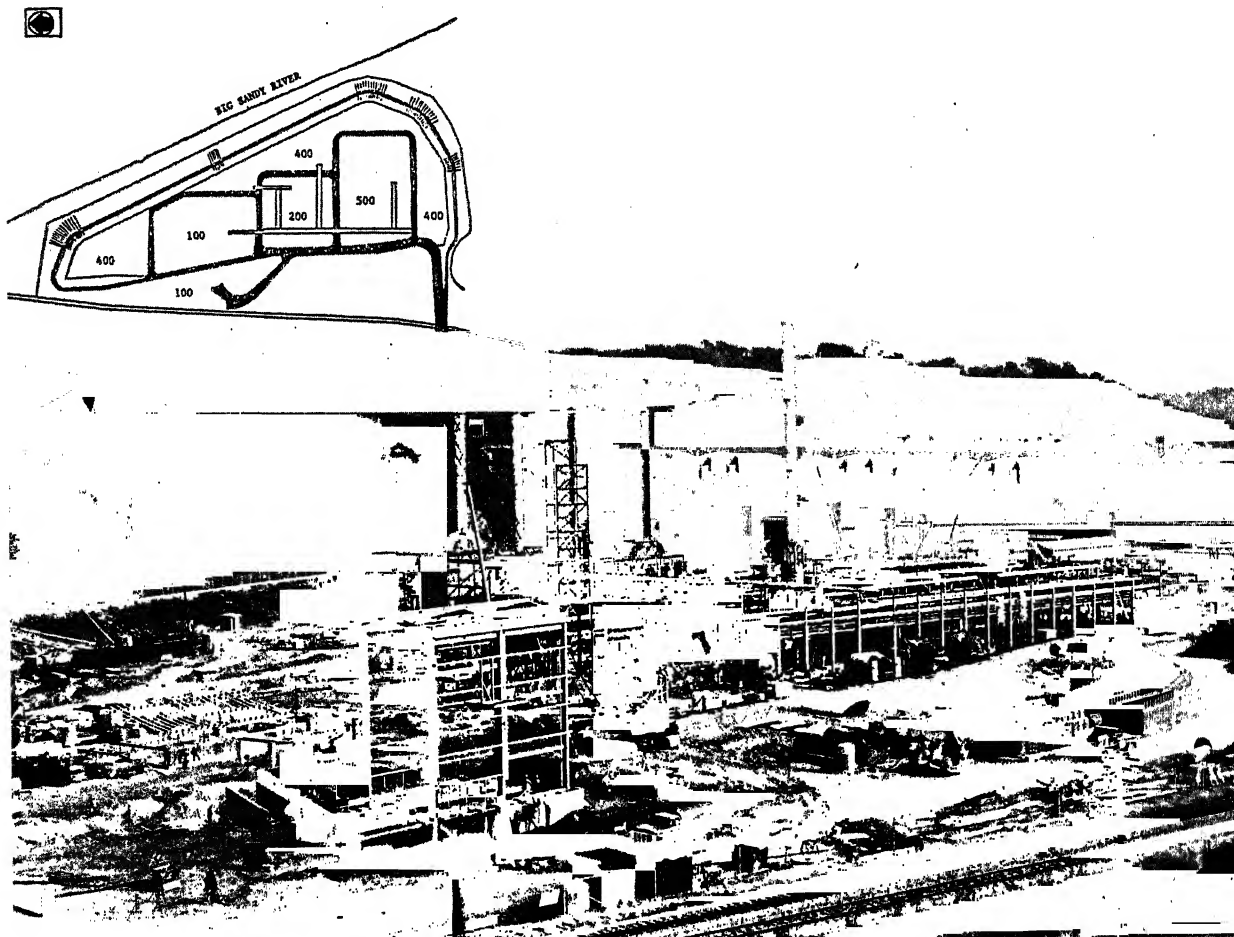
**RECENT WORK AND ACCOMPLISHMENTS** — The final design and engineering work required for construction has been nearly completed by Hydrocarbon Research, Inc., Miami. Ashland's efforts have been concentrated on coordinating and supplementing the HRI design effort, obtaining the necessary environmental permits for construction and operation of the pilot plant, and managing construction of the pilot plant. The subcontractor for construction of the major percentage of the pilot plant (Badger Plants) entered the field in March 1977. Their construction effort was 27 percent complete as of September 30, 1977. Work has concentrated on the coal preparation section, hydrogenation and reactor effluent section, tank storage area, and wastewater treatment areas within the pilot plant. The Chemical Storage Building is complete and the erection of other support buildings is continuing as scheduled. The Administrative Building will be complete in early 1978. ASFI negotiated a subcontract with C.E. Lummus for design and engineering of the solids/liquids separation section of the plant. Their method for solids/liquids separation depends on an antisolvent deashing mechanism. Lummus has completed approximately 35 percent of the engineering for the project.

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\*Phases II and III funding participants are Ashland Synthetic Fuels, Inc., Commonwealth of Kentucky, Conoco Coal Development Co., Electric Power Research Institute, Mobil Research and Development Corp., and Standard Oil of Indiana.

\*\*The total \$164.1 million includes funds for Phase II laboratory support and pilot plant equipment purchases.

**PLANS FOR THE COMING YEAR** — Construction of the plant is scheduled for completion in the fourth calendar quarter of 1978. Checkout will begin in late spring and continue throughout the year. ASFI will work during the year to acquire all remaining necessary permits for operation of the plant. The operations training program, already underway for the supervisors, will begin for hourly personnel. The contract for construction of the Lummus antisolvent deashing unit will be negotiated in 1978. Construction of the unit will be completed in late 1978.



*H-Coal Plant: Coal Receiving Hopper in Lower Left*

## EBULLATED-BED FLUID DYNAMICS FOR H-COAL PROCESS

AMOCO OIL COMPANY  
DOE - \$792,000  
8/22/77 - 8/22/79

**OBJECTIVES** — This project is seeking to improve the control of the H-Coal ebullated-bed reactor through a better understanding of the hydrodynamics of ebullating beds. Specific objectives are to review prior work on ebullated beds, design and construct a cold-flow unit and collect data on ebullated beds, and develop a mathematical model. The mathematical model will be aimed at defining the steady-state ebullated-bed conditions as a function of operating parameters and development of criteria for bed stability.

**RECENT WORK AND ACCOMPLISHMENTS** – The review of prior work has been completed, including the areas of gas-liquid-solid fluidization, liquid-solid fluidization, vertical gas-liquid flow (bubble and slug behavior), experimental techniques in multiphase flow, and properties of coal-oil mixtures. The process design of the cold-flow unit has been completed also. Major pieces of equipment including the gas compressor, feed tanks, mixers, gas-liquid separator, piping, and support structure have been ordered. A computer capable of running experiments on the unit and taking and analyzing data has been selected. Experimental techniques to measure the holdup of the individual phases in the bed have been selected. A survey of laboratories capable of measuring the viscosity and surface tension of H-Coal slurries under H-Coal reactor conditions has been completed.

**PLANS FOR THE COMING YEAR** – A report documenting the review will be completed, and a related report will be issued in the first quarter of 1978. Construction of the cold-flow unit will be completed, and experiments on liquid-solid and gas-liquid-solid fluidization will begin. Data will be analyzed to test the validity of the mathematical model of three-phase fluidization systems proposed in the literature. The experimental program will be consistent with the needs of the H-Coal program. A laboratory will be selected to measure the viscosity of H-Coal slurries under reactor conditions. Samples will be taken at HRI's PDU in Trenton, New Jersey, and transferred to a selected laboratory for the measurements.

## **SYNTHOIL PROCESS DEVELOPMENT**

### **PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$3,000,000**

**1969 - Continuing**

**OBJECTIVES** – The near-term goal of this coal hydroliquefaction project is to continue development and complete acquisition of scaleup and operations data for testing in a larger size PDU. The distinguishing feature of this process is its unique fixed-bed turbulent-flow reactor system, which converts coal into a clean liquid fuel by rigorous reactant contacting through rapid flow of hydrogen.

**RECENT WORK AND ACCOMPLISHMENTS** – Stable long-term operability of the process was demonstrated by a run of 1068 hours in the 1/2-t/d Synthoil-I unit. During this run, the plant was shut down, placed on standby, and then restarted to demonstrate that the process can be controlled effectively. In addition, this run showed that an added catalyst is not required; nearly complete liquefaction is accomplished by the turbulent contacting in the reactor coupled with self catalysis by the indigenous minerals in the coal. The product was ambiently liquid and satisfied air pollution restrictions on sulfur content.

The preheater optimization study was completed and resulted in a configuration that has less than 60 percent of the volume of the original design, which considerably reduces the estimated preheater construction cost. Experimental work also showed that double centrifugation (a batch

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to the extent that solids removal in the continuous mode is significantly better than that achieved by double processing in the batch centrifuge.

Fabrication of the fixed-bed turbulent-flow reactor cold model for heat transfer studies was completed and shakedown tests run. Experiments defined effective thermal conductivities and wall heat transfer coefficients at conditions simulating those in an operating fixed-bed reactor. Work also progressed on defining the flow regimes encountered in two-phase upflow in the reactor. Work on the mathematical model of the Synthoil process design was initiated. This model will be used to conduct optimization studies and to provide design data for large-scale hardware.

Construction of the new 1-t/d Synthoil-II unit is well along. About 20 percent of the installation effort has been completed; all components have been procured.

**PLANS FOR THE COMING YEAR** — Since it has been determined that no catalyst is required for the Synthoil process, further development will be restricted to confirmation runs to obtain baseline data. Facilities will be converted to use in broader process studies during the year. Several long-duration runs will be made with West Virginia and Western Kentucky coals in the 1/2-t/d Synthoil-I unit to further demonstrate long-term operability and the uniformity of product quality over extended time periods with improved plant components. Effects of increased throughput will be evaluated. The continuous centrifuge will be totally integrated into operations with the 1/2-t/d unit. Construction of the next 1-t/d unit will be completed to produce synthetic oil for upgrading. Supporting research in the areas of novel solids separation, heat transfer, and process modeling will continue.

## **SYNTHOIL 10-T/D PROCESS DEVELOPMENT UNIT**

**FOSTER WHEELER ENERGY CORPORATION**

**DOE - \$24,425,676**

**1974 - 1978**

**OBJECTIVES** — This work involves the design and construction of a 10-t/d PDU based on the Synthoil process design to provide scaleup and design data for commercial plants that convert coal to nonpolluting fuel oil.

**RECENT WORK AND ACCOMPLISHMENTS** — The Synthoil process produces a clean synthetic boiler fuel oil from various high-sulfur bituminous coals. A portion of the whole product oil, without fractionation, is recycled to mix with dried ground coal as a slurry. Hydrogen is added to the slurry and the mixture heated prior to entering the fixed bed reactor. Operating temperature is about 850°F, and the pressure is 4000 psig. The reactor is packed either with commercial catalyst pellets of cobalt molybdate or with inert pellets. Reaction gases are removed and cleaned and the solids separated from the liquid oil by continuous centrifuging. A large volume of hydrogen is recycled with the slurry through the fixed bed so as to provide extreme turbulence. This turbulence must prevent catalyst deactivation and bed plugging if the fixed bed configuration is to be practical.

Supporting research at PERC has indicated process operability with inert pellets replacing the catalyst over extended periods at a relatively low coal throughput rate. Plans include comparing results using inert pellets in the fixed bed with thermal reactions in an empty reactor, attaining



maximum throughput of coal, using a continuous centrifuge to provide process solvent, developing techniques to remove deposits from the packed bed, and other support studies.

**PLANS FOR THE COMING YEAR** – The PDU construction project is 87 percent complete. The mechanical completion date has slipped from September 1977 to June 30, 1978, but the project remains within budget. There are no plans to operate the PDU in the Synthoil mode. Plans are to place the unit in standby to await identification of an appropriate third-generation process for testing and suitable funding arrangements for such work.

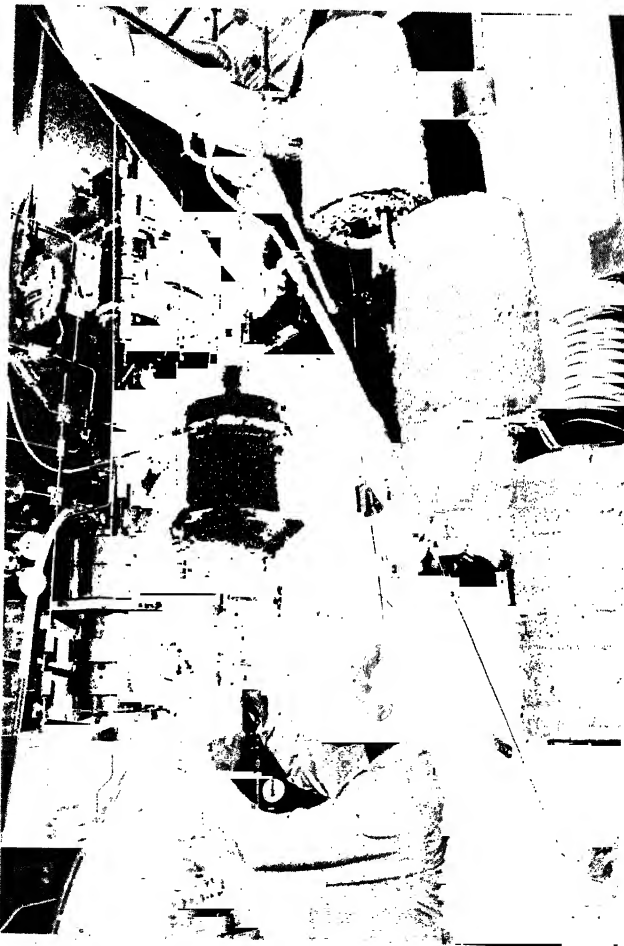
### CHEMICAL STUDIES ON SYNTHOIL PROCESS

SANDIA LABORATORIES

DOE - \$620,000

9/1/75 - 9/30/77

**OBJECTIVES** – This research program was designed to support the Synthoil process for coal liquefaction. It involved three separate tasks: mechanisms of catalyst deactivation, effects of mineral matter on coal liquefaction, and preheater processes. The results of these studies are to be applied to existing unit operation for improvement in catalyst lifetime and overall process operation.



*Batch Autoclave for Studying Mineral Matter Effects on Coal Liquefaction*

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**RECENT WORK AND ACCOMPLISHMENTS** – Catalyst studies indicated the major deactivation mechanism is coking, which occurs rapidly. Subsequent titanium and nitrogen poisoning continue to decrease the catalyst activity. Also, catalyst at the exit of the Synthoil reactor showed more deactivation than the catalyst at the entrance to the reactor. Recommendations to increase catalyst lifetime included using a less acid catalyst support and modifications in operating procedures.

Mineral matter effects were studied by batch hydroliquefaction of high-volatile bituminous coals: with similar petrographic (maceral) composition but different mineral contents; with minerals chemically and physically removed; and with mineral matter physically added. Statistical analyses of the data, for coals with mineral contents between 5 and 23 weight percent, have shown that pyrite had a significant effect on conversion to benzene solubles, conversion of preasphaltenes to pentane-soluble oil, and reduction of product viscosity. Reduced iron sulfide, which is derived from the pyrite as a result of the liquefaction process, was found to have a significant effect on both conversion and desulfurization. Other naturally occurring minerals in coal (quartz, calcite, kaolinite, montmorillonite) did not show nearly as much, if any, catalytic activity.

A continuous reactor with four independently controlled stages was set up to simulate the Synthoil preheater. Slurries of West Virginia coal in creosote oil were reacted at 4000 psi, 430°C in the packed reactor. Decrease in pre-asphaltenes and viscosity occurred through the reactor with co-current increases in pentane soluble oils and gases. Asphaltene content remained reasonably constant. Regression analyses were used to correlate viscosity, conversion, and other product characteristics with liquid space rates and hydrogen flow rates.

**PLANS FOR THE COMING YEAR** – The program in support of Synthoil is complete and is being documented. Studies on catalyst deactivation mechanisms in the H-Coal process, quantification of mineral effects, and continuous-reactor (noncatalytic) coal conversion studies will be carried out under other programs in 1979.

## PHYSICAL PROPERTIES OF SYNTHOIL PRODUCTS

BATTELLE, COLUMBUS LABORATORIES

DOE - \$155,267

6/75 - 8/77

**OBJECTIVES** – This program examined certain properties of Synthoil liquids: viscosity, resistivity, dielectric constant, and density, together with the relationships among them. Of particular interest were the dependence of these properties on pressure and temperature. The determination of viscosity, and especially its dependence on shear rate, required the use of novel techniques. The results are important in connection with the design of equipment for liquefaction and for subsequent solids separation.

**RECENT WORK AND ACCOMPLISHMENTS** – The apparatus used for viscosity and resistivity includes a cylindrical vessel with a cylindrical bob, operated axially in the liquid. The bob was connected to a permanent magnet. Outside the pressure vessel, coils sensed the motion of the bob assembly and, by means of a controlled current, caused it to rise and fall. Heaters maintained a constant ( $\pm 1^\circ\text{C}$ ) temperature. Through calibration, the rate of shear and the shear stress could be determined. The properties of coal liquids depend on many factors, not the least of which are the source of the coal and the conditions of liquefaction. Only the dependence on temperature, pres-

sure, and solids content were examined. Results for the viscosity of a product liquid showed that they displayed the properties of a Bingham liquid; the shear stress was linear with shear rate, intercepting the axis at a non-zero value of yield stress. A linear relationship was observed between the pressure and temperature coefficients of viscosity. Electrical resistivity and dielectric constant were measured in the same apparatus by using the insulated bob as one electrode, the pressure vessel as the other. Results for resistivity were qualitatively similar to those for viscosity.

**PLANS FOR THE COMING YEAR** – The property determinations were completed and the program terminated.

### **ZINC HALIDE HYDROCRACKING PROCESS**

CONOCO COAL DEVELOPMENT COMPANY (CONTINENTAL OIL CO.)

DOE - \$10,269,372; Conoco Coal/Shell Development - \$1,141,041

1/75 - 5/80

**OBJECTIVES** – The zinc halide process is being developed to produce clean gaseous and liquid fuels from coal, with particular emphasis on gasoline. The work includes continuous bench-scale and PDU investigation of zinc halide as a catalyst for the hydrogenation and hydrocracking of coals and coal extracts, and development of an economical regeneration process for the efficient recovery of zinc halide from the spent melt. Appropriate economic studies will be made to indicate the areas in which this process can reduce the need for imported petroleum.

**RECENT WORK AND ACCOMPLISHMENTS** – The continuous bench-scale program was completed, providing data on kinetics and yields for liquefaction of subbituminous coal in molten zinc chloride. This work included a program extension to test two reactors in series, recycle of distillate oils, and high-temperature stripping. A correlation was developed to predict conversion of Colstrip subbituminous coal as a function of time at temperatures between 385° and 427°C (725° and 800°F) and hydrogen pressures between 13.9 and 20.8 MPa (2000 to 3000 psig). Within this range, the effect of pressure was negligible. An indication of the catalytic nature of zinc chloride liquefaction is that substantially all of the yields of hydrocarbon gases and liquids could be correlated by a single line against conversion; that is, these yields are independent of the temperature used (within the range tested).

The gasoline-range naphtha produced had a Research Octane Number of 91 to 92 and a Motor Octane Number of 79 without further processing. Higher-boiling distillates have unusually low sulfur and nitrogen values, and if not converted on recycle, would be excellent fuel oils. Experimental tests on regenerating used catalyst from processing Colstrip coal indicated that under recycle conditions, 98.2 percent of the zinc chloride fed would be recovered, with 1.8 percent being retained in the ash collected via hot cyclone. Batch and semicontinuous tests in a fluidized bed have defined conditions and recoveries for secondary recovery of zinc and iron from this ash. Overall recovery of zinc can be raised to 99.6 percent by secondary treatment with air and HCl at 1038°C (1900°F) and atmospheric pressure. A study of the economic incentive for such secondary treatment showed expected savings of 3.0 to 3.6 cents/gal of gasoline in addition to environmental advantages; therefore, this step will be added to the process.

Construction of the 100 lb/hr PDU has proceeded substantially on schedule, with the feed and high-pressure hydrocracking sections completed and ready for pressure testing on December 1,

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1977. The remainder of the liquefaction section will be completed in May 1978. Supporting batch work has shown the amenability of other coals to zinc chloride processing and provided special equipment for testing densities and viscosities of used zinc chloride catalyst.

**PLANS FOR THE COMING YEAR** – The main emphasis will be on breaking in the new PDU. Initially, the liquefaction section will be operated separately on SRC from Ft. Lewis. The regeneration section will be completed by May 1978 and, after the hydrocracking program (June), be broken in by feeding the used catalyst from liquefaction runs. Thereafter, alternating operation of the two halves will be used to obtain material balance data. Subbituminous coal will then be fed, and alternating operation followed by integrated, simultaneous operation of the entire PDU with coal feed.

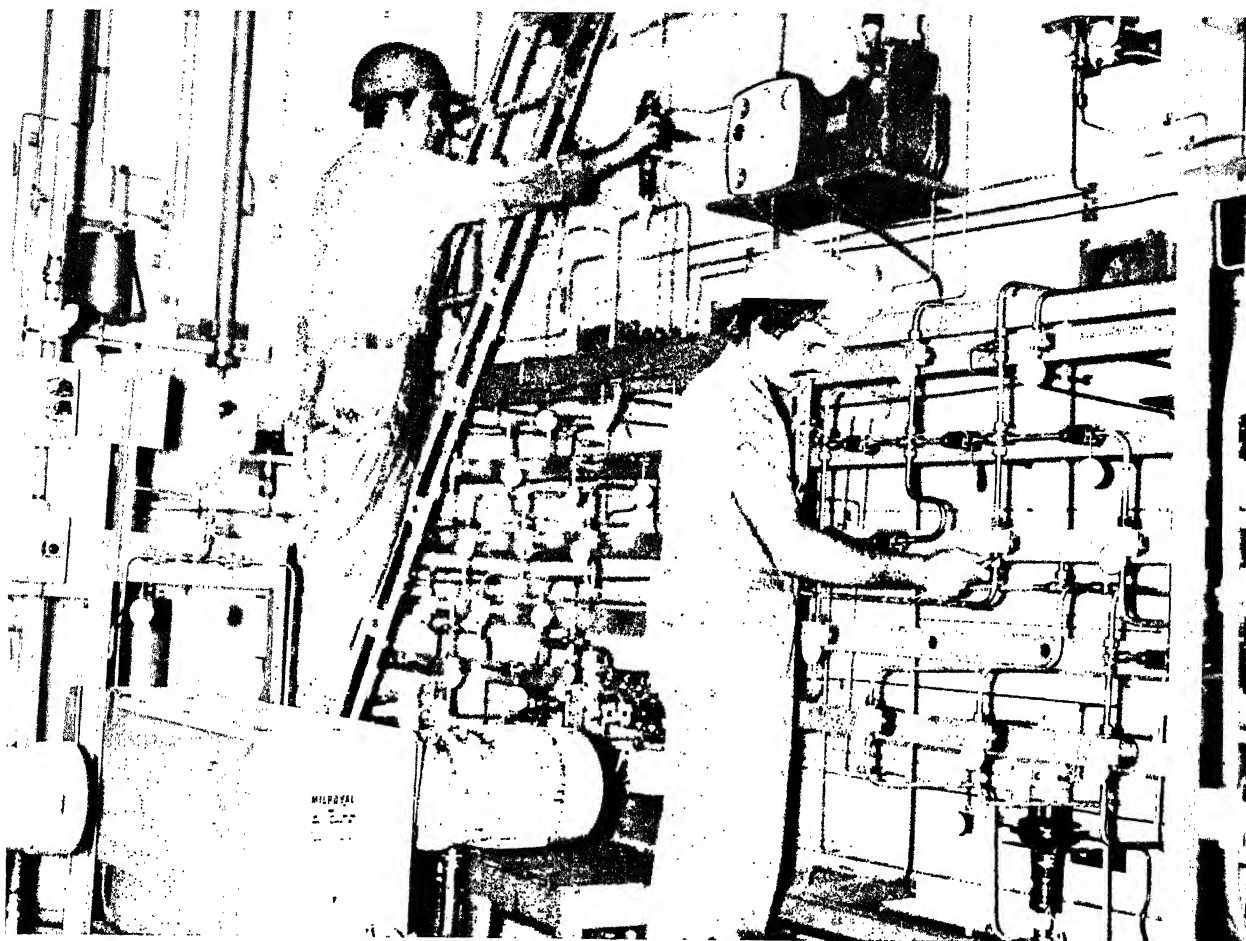
## HYDROLIQUEFACTION USING DISPOSABLE CATALYSTS

PITTSBURGH ENERGY RESEARCH CENTER  
DOE - \$601,000  
1976 - Continuing

**OBJECTIVES** – This project is an extension of the technology employed in the Bergius process that operated on a large scale in Germany during the 1940's. A substantial part of the technology and equipment is considered usable today; however, the original process had several deterrents impeding commercial implementation under current economic conditions such as high-pressure (10,000 psi), long-reactor residence time, and limited removal of sulfur compounds. Current R&D efforts are directed toward improving the economics of the original process while simultaneously producing a low-sulfur fuel. Advanced reactor designs that provide maximum gas-liquid-solid contacting eliminate hydrogen starvation. New combinations of catalysts that promote liquefaction as well as desulfurization have the potential to reduce the operating pressure fivefold and to increase the throughput by a factor of six—depending upon the reactivity of the particular coal used thus improving the process economics considerably.

**RECENT WORK AND ACCOMPLISHMENTS** – Several experimental units are being operated or constructed for this project. The 20-lb/hr continuous stirred-tank unit was operated for a total of 1200 hr while feeding Western Kentucky coal. Runs were made at 450°C, 2000 and 3000 psi, without added catalyst; at 450°C, 2000 and 3000 psi, with 1 weight percent iron as ferrous sulfate added to the coal by mixing in aqueous solution and drying; and at 450°C, 3000 psi, using 1 weight percent iron as ferrous sulfate dry blended with the coal. Results of these experiments showed essentially no difference in the benzene-soluble oil yield between the runs with no added catalyst and those where ferrous sulfate was used. At 2000 psi, the benzene-soluble oil yield was about 60 weight percent of the moisture and ash free coal feed, which increased to 65 weight percent at 3000 psi. Sulfur content of the oil was attractively low, ranging between 0.2 and 0.6 weight percent.

Construction began on the 1/2-t/d PDU early in the year and has progressed to 90 percent completion. All plant piping is complete, and nearly all pressure vessels are in place. Major remaining items include electrical wiring, heaters, instrumentation, steam tracing, and insulation. Construction and shakedown testing of the hot-charge batch autoclave unit, to obtain kinetic data on the various disposable catalysts, were completed. A baseline run was made using 40 weight percent Western Kentucky coal in a high-boiling fraction of creosote oil without added catalyst at the conditions employed in the 20-lb/hr unit: 450°C and 3000 psi.



*New 1-T/D Disposable Catalyst Liquefaction Unit Nearly Complete;  
Technicians Complete Assembly Of High-Pressure Piping and Control Valves  
for Gas Recirculation and Gas and Liquid Product Depressurization Systems*

**PLANS FOR THE COMING YEAR** — During the first quarter, construction of the 1/2-t/d PDU will be completed, and pressure testing will begin followed by shakedown operations. Experiments with and without catalyst will be conducted in both the 1/2-t/d and 20-lb/hr units to evaluate the effectiveness of various reactor configurations and the desulfurization activity of several disposable catalysts. Testing of catalysts in the batch autoclave will continue; these experiments will establish the relative desulfurization activity of various disposable catalysts as well as precise kinetic information. An existing 1-lb/hr coal-slurry hydrogenation unit, with a reactor section consisting of a length of 5/16-inch ID tubing packed with inert glass pellets, will be modified to evaluate a large variety of conditions to optimize process variables, with good gas-liquid contacting for the promising disposable catalysts, before testing in the larger units.

#### CLEAN INDUSTRIAL AND TRANSPORTATION FUELS FROM COAL

THE LUMMUS COMPANY  
DOE - \$4,790,809; Lummus - \$1,301,000  
9/28/76 - 8/30/78

**OBJECTIVES** — This program seeks to establish conditions of optimum and reliable operations for the Lummus Clean Fuel from Coal (CFFC) Process operating in an all-distillate mode; specifically,

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to establish recycle paste oil self-sufficiency, catalyst life, process operability for coal liquefaction, and demonstrate comparative hydroliquefaction performance of the initial nickel-molybdenum catalyst versus a standard cobalt-molybdate catalyst; convert the all-distillate coal liquid product to gasoline by continuous fixed-bed hydrotreating/hydrocracking operations; and provide a preliminary engineering design of a Lummus CFFC pilot plant facility. Successful completion of this program will confirm both a route for the conversion of coal to a low-ash, low-sulfur, low-nitrogen all-distillate industrial boiler fuel, and its subsequent conversion to gasoline. The antisolvent de-ashing technology used in this program offers a viable method of ash removal that reduces both capital and operating costs when compared with processes based on filtration or centrifugation. Further, the antisolvent method is generally adaptable to other liquefaction processes. This feature together with the coal hydroliquefaction using expanded beds with no internal recycle provide the basis for a more economical coal-based route to either clean industrial fuel or to gasoline and other transportation fuels.

**RECENT WORK AND ACCOMPLISHMENTS** – Subcontractor mechanical and electrical installation work on integrating the CFFC pilot plant facility were completed. The facility consists of an expanded-bed coal hydroliquefaction unit, an antisolvent de-ashing module, a vacuum flash unit for de-asher underflow stripping, a vacuum flash unit for the deep flashing of the feed for the hydrofining/hydrocracking operation, a continuous distillation unit, and a number of ancillary storage and run tanks needed to sustain integrated operation. Each of these individual units has been run and debugged, and the entire facility has been operated on a continuous and integrated basis. The preparation of equilibrium recycle paste solvent is well advanced. The bench-scale hydroliquefaction catalyst activity testing work was completed. On the basis of these tests, a determination was made to utilize a commercially available nickel/molybdenum catalyst for the paste solvent self-sufficiency subtask.

**PLANS FOR THE COMING YEAR** – Activities will include completion of the demonstration of recycle paste solvent self-sufficiency, confirmation of catalyst life data, tests of high-space velocity operation on the liquefaction reactors to establish limiting conditions related to the effects of high viscosity on catalyst bed expansion in the first reactor, completion of comparative test work on a standard cobalt-molybdate catalyst, conversion to gasoline of a portion of the net all-distillate coal liquefaction product by hydroprocessing in a fixed-bed unit, and start of the preliminary engineering design work of a Lummus CFFC pilot plant facility. The latter two tasks will be initiated if authorized by DOE.

## **PARTIAL LIQUEFACTION OF COAL BY DIRECT HYDROGENATION**

**ROCKWELL INTERNATIONAL, ROCKETDYNE DIVISION**

**DOE - \$4,327,490**

**8/1/75 - 9/1/79**

**OBJECTIVES** – The goal is to develop and demonstrate a reactor system as part of a process for partial liquefaction of coal in which coal is reacted with hydrogen in a high-mass-flux short-residence-time reactor. The process is based on a concept, often termed flash hydropyrolysis, that high-liquid yields are favored when coal is allowed to react with hydrogen for a brief period followed by rapid quenching. It appears to have several advantages over solvent- or slurry-based concepts. A much smaller reactor is required, and no catalyst is needed. Dry feeding of coal is used,

which eliminates the need for solvent recycle, slurry pumping, and paste preparation. A low-sulfur liquid product is recovered in the vapor phase, so that no additional char/product separation is required.

**RECENT WORK AND ACCOMPLISHMENTS** – Reactor testing was begun in a 1/4-t/hr unit and was continued in a 1-t/hr unit. In all, 21 1/4-t/hr tests were made, and 35 1-t/hr tests were completed by the end of November 1977. Test durations up to 1 hr with up to 1500 lbm of coal being processed in a single test have been achieved. Tests have been made over a reactor temperature range from 1100° to 1950°F, a pressure range from 500 to 1500 psig, and a reactor residence time range from 30 to 640 milliseconds. In the 1-t/hr unit, the reaction chamber ranges in size from 2.37- to 4.87-in. ID by 5.0-ft long. The reactor system, including the coal feeder, have functioned well with caking western Kentucky coals. During the first 2 years of process development, the following results have been achieved: routine operation with caking western Kentucky coals without pretreatment, vapor-phase liquid product recovery with no filtration or distillation required, up to 11 percent carbon conversion to BTX, a fine, dry char suitable for direct feeding into a gasifier for H<sub>2</sub> production, a versatile reactor configuration that can be adjusted to maximize either liquid or gas production, a low-sulfur liquid product (0.3 percent), high utilization of hydrogen contained in coal so the consumption of free hydrogen is low, development of a highly successful dense-phase coal feeding method, and 94 percent carbon recovery in short-duration tests.

**PLANS FOR THE COMING YEAR** – The 1-t/hr reactor testing will be continued and equipment will be added to the PDU to allow processing up to 3½ tons of coal in a single test and to allow better product separation and recovery. Also, preliminary economic evaluation of the process will begin.

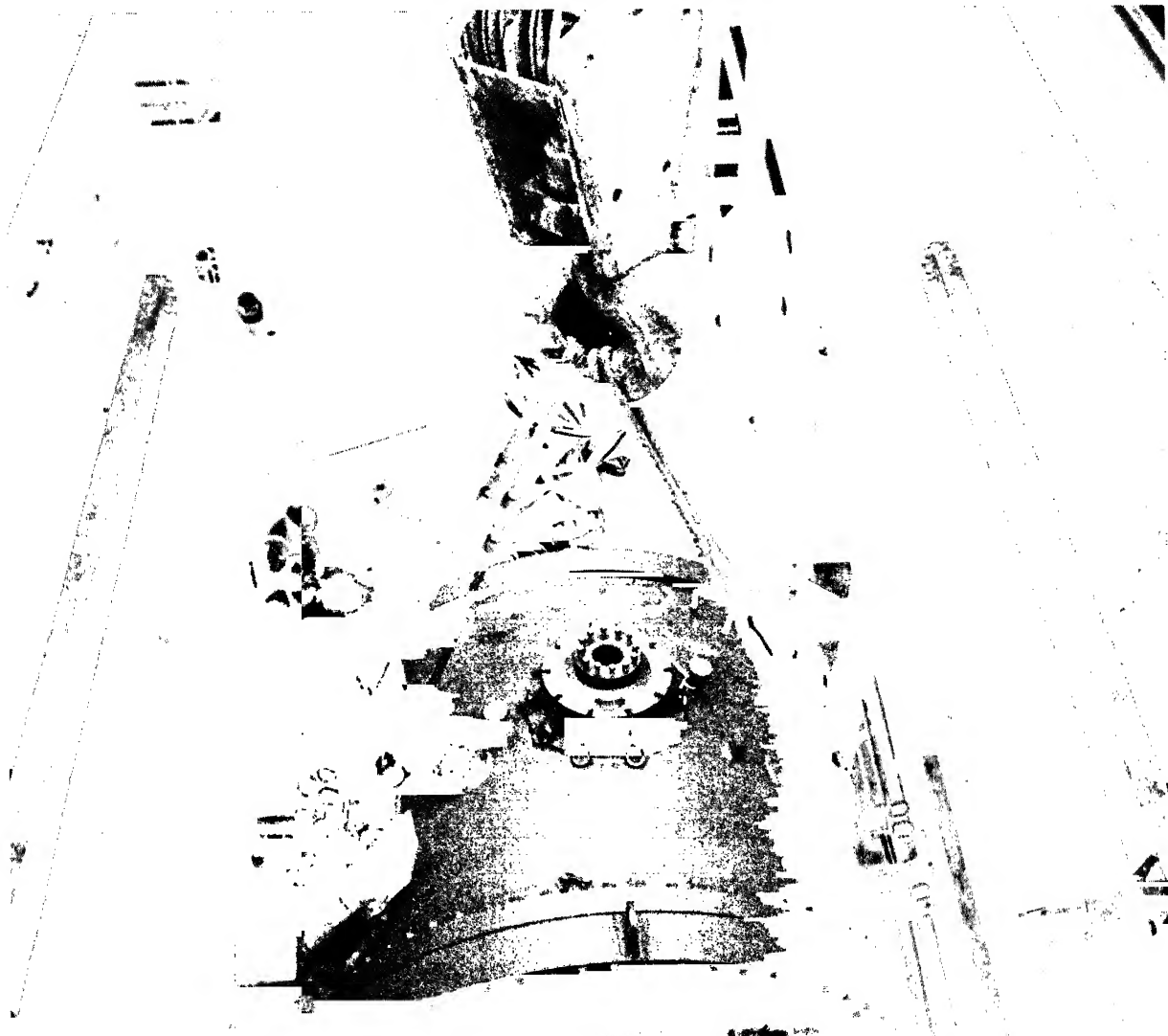
### **SOLVENT REFINED COAL PROCESS**

THE PITTSBURG & MIDWAY COAL MINING CO.  
DOE - \$88,760,000  
10/66 - 12/78

**OBJECTIVES** – The goal of this project is to successfully establish in a pilot plant the conversion of coal to low-ash, low-sulfur fuels. This work will include process development, the evaluation of process equipment, bench-scale testing to provide data for guidance of pilot plant operations and for use in cost studies and commercial-scale process designs. A major corollary objective is the production of sufficient volumes of Solvent Refined Coal (SRC) products for meaningful consumer testing.

**RECENT WORK AND ACCOMPLISHMENTS** – Major accomplishments at the SRC pilot plant during the period include: completion of the 3000-ton production commitment of solid SRC for combustion testing, modification of the pilot plant to operate in SRC-II mode producing a distillate fuel, and startup and successful operation of the pilot plant in the SRC-II mode. Record production for any month (500 tons) or any quarter (1215 tons) of SRC-I operation was achieved during the fourth quarter of 1976. A record 61 days of continuous operation and a record 83.7 percent on-stream factor for a quarter were achieved during the third quarter of 1977, while operating in the SRC-II mode. Additional work accomplished at the pilot plant includes: a process variable study of rotary precoat filtration, successful SRC-II operation with Kentucky Nos. 9 and 14 and Illinois No. 6 coal, the completion of nine material balance runs in the SRC-II mode, and an analysis and

correlation of process variable effects in the processing of Kentucky Nos. 9 and 14 and Illinois No. 6 coal. Engineering and construction for installation of the Johns-Manville experimental rotary precoat filter are well underway, and preparations for the Lummus antisolvent deashing system have begun.



*New EPRI Rotary Filter Being Installed*





*SRC Plant: Coal Conversion and the Environment Can Co-Exist*

Major accomplishments at the P&M Research Laboratory in Merriam, Kansas, were the completion of an extensive process variable study on SRC-II processing of Kentucky Nos. 9 and 14 coal, exploratory operation with three Pittsburgh seam coals, exploratory operation with Illinois No. 6 coal, and exploratory operation with Amax subbituminous coal. The Illinois coal and two of the Pittsburgh seam coals were found to be suitable for processing in the pilot plant. The third Pittsburgh seam coal was found to be too deficient in mineral matter, and the Amax coal was found to generate carbonate solids. An analysis and correlation of the process variable effects in the SRC-II processing of Kentucky Nos. 9 and 14 coal was published, and the study was extended to lower hydrogen feed rates and to differing hydrogen partial pressures. Preliminary exploratory experiments were conducted to determine the effect of reactor configuration on process operability and yields.

Environmental monitoring continued around the SRC pilot plant. Results show no measurable deterioration of the environment. Preparation of a detailed report on the baseline and plant operating environmental studies was begun. Trace element studies were continued by Washington State University, and samples were obtained to measure trace element distribution in the SRC-II mode of operation. A toxicology program based on animal bioassays was begun. Eight product and intermediate streams from the SRC process are being examined.

**PLANS FOR THE COMING YEAR** — The major emphasis at the pilot plant during the coming year will be on development of mineral separation technology. The Johns-Manville experimental filter installation will be completed, and equipment and process trials conducted. The Lummus antisolvent deashing unit will be installed and initial startup made late in the period. Process variable studies in the SRC-I and the SRC-II modes will be made with additional coals. The Merriam Laboratory will support the process variable studies at the pilot plant by conducting exploratory trials in the SRC-I and II modes. Later, emphasis will be placed on process improvements by changes in the reactor configuration, reaction conditions, and the use of additives. The environmental monitoring trace-element distributions studies, and the toxicological program will continue.

#### **SRC PILOT PLANT, WILSONVILLE, ALABAMA**

**SOUTHERN COMPANY SERVICES, INC.**

**DOE - \$5,740,892; Electric Power Research Institute - \$2,988,000**

**1/1/76 - 12/31/77**

**OBJECTIVES** — This program seeks to broaden the data base for design and economic evaluation of commercial SRC plants. Specific objectives for 1977 were to evaluate: three industrially significant coals of various ranks, and improvements in solid-liquid separation methods, such as centrifuges and antisolvent deashing.

**RECENT WORK AND ACCOMPLISHMENTS** — The three coals investigated in 1977 included a subbituminous coal from Wyoming and bituminous coals from Utah and Indiana. From each coal, SRC was produced that met the EPA new source performance standards for sulfur dioxide and particulates. The Wyoming coal was the most difficult to process and yielded the least SRC. Inorganic solids produced from the coal settled in process vessels and caused operating problems. A system was added to the dissolver that will facilitate removal of these solids from the vessel. New distillation equipment was added to improve the separation of the various liquid products and to improve the consistency of process solvent composition. Centrifuges and an antisolvent deashing

system were evaluated as alternatives to filtration for mineral residue separation. The preliminary results from these tests indicate that centrifuges or antisolvent deashing did not produce SRC with an ash content as low as that which could be produced by filtration. No economic evaluation was conducted.

**PLANS FOR THE COMING YEAR** — Work will continue on the problem of inorganic solids accumulating in the dissolver and their effect on process yields. The scope of solid-liquid separation tests will be expanded to include other processes that may be more economical, including an evaluation of a critical solvent deashing process developed by Kerr-McGee corporation.

### **FULL-SCALE UTILITY BOILER TEST WITH SRC**

SOUTHERN COMPANY SERVICES, INC.

DOE - \$1,217,157

6/30/76 - 3/31/78

**OBJECTIVES** — The objective of the SRC burn test program is to demonstrate the advantages of SRC as a boiler fuel by investigating its handling and storage features, its burning characteristics, and its ability to comply with existing EPA emission standards. Attainment of this objective will show that a commercial boiler and its auxiliary equipment can be modified to handle SRC, and that the use of this fuel offers a means for pollution abatement at existing coal-fired units. Further, it is anticipated that the use of SRC will lead to a reduction in plant ash and sludge handling facilities, higher overall thermal efficiency, and lower maintenance requirements.

**RECENT WORK AND ACCOMPLISHMENTS** — The SRC combustion test was performed in three phases at Plant Mitchell, operated by Georgia Power Company near Albany, Georgia. Phase I was to operate and test a 22.5 Mw, pulverized-coal-fired boiler under normal operating conditions firing coal. For Phase II, new burners and pulverizer feeders were installed, and the boiler was tested again firing coal. During Phase III when SRC was fired, hot air to the pulverizer was blanked off, pulverizer spring pressure was reduced, and the boiler was tested for a third time. Tests and measurements performed during each phase of the test program included: boiler efficiency; emissions tests using EPA and ASTM procedures for particulates,  $\text{SO}_2$ , and  $\text{NO}_x$ ; and continuous gas analyzers that operated 24-hr/d monitoring the flue gas for opacity,  $\text{SO}_2$ ,  $\text{NO}_x$ ,  $\text{CO}_2$ , and  $\text{O}_2$ .

Prior to beginning Phase I testing, Plant Mitchell Unit 1 was brought off line in January, for inspection and repair of the boiler, pulverizers, fans, precipitators, and other auxiliary equipment. Instrumentation installation and calibration, adjustments, and various modifications were carried out. Phase I testing was then initiated on March 22 and completed on March 30, 1977. Six boiler efficiency tests—two each at full, medium, and low load—were conducted. A series of nine emissions tests was performed at both the inlet and the outlet of the precipitator: three tests at full, medium, and low loads, respectively. April and May 1977 were devoted to retrofitting the unit for Phase II tests, which were conducted from May 24 to June 1, 1977. Five boiler efficiency tests—two at full load, two at medium load, and one at low load—emissions tests, and other tests were conducted as planned. Prior research had shown that variable-speed fuel feed, cold primary air, and reduced pulverizer ball pressure loading were necessary modifications for grinding SRC in ball and race type pulverizers. This work and various other maintenance tasks were completed, and Phase III testing began on June 13, 1977. Boiler efficiency and emissions tests were conducted as in the previous

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phases of testing. During these tests, SRC demonstrated exceptional characteristics as a boiler fuel. Ignition was easily initiated, flame stability was excellent, and the boiler operated continuously for 18 days with no problems. This boiler normally requires soot blowing from 6 to 12 times a day; however, during the 18-day test burn of SRC, soot blowing was not required at all. The quantity of flyash generated when firing SRC was nominally 7 to 10 times less than when firing coal, and bottom ash was virtually nonexistent. The pulverizers were operated under many conditions related to fuel-air ratio, fuel bed depth in the pulverizer, and ball loading, and no problems with pulverizing SRC were encountered. Pulverizer capacity was at least 25 percent greater with SRC than with coal, when compared on a Btu basis.

Preliminary data indicate that SRC easily complies with existing EPA New Source Performance Standards for SO<sub>2</sub> and NO<sub>x</sub>. Particulate concentrations leaving the primary precipitator were higher than EPA standards; however, this is attributed to the precipitator which was installed in 1946 and is obsolete by current design standards. Since the unit is equipped with a secondary precipitator of modern design, additional tests were performed using this precipitator. These tests indicate EPA compliance by a wide margin and show that an adequately designed precipitator enables SRC to meet existing EPA particulate emission standards.

**PLANS FOR THE COMING YEAR** – Data retrieval and analysis is continuing, with results to be published in a comprehensive report during the first quarter of 1978.

## DEMONSTRATION OF SRC AS A GAS TURBINE FUEL

ENCOTECH, INC.  
DOE - \$440,424  
6/17/76 - 11/1/78

**OBJECTIVES** – This project is designed to demonstrate the suitability of Solvent Refined Coal (SRC) as a liquid-fueled boiler and gas turbine fuel. The work encompasses process development in laboratory batch washing tests, design and fabrication of a pilot SRC processing skid, and operation of a small-scale atmospheric-pressure combustion chamber on processed SRC fuel. The successful demonstration of SRC as a gas turbine fuel will pave the way towards commercial utilization of this coal-derived fuel in gas turbine installations, and ultimately as a power-generation fuel in highly thermal-efficient combined-cycle (steam and gas turbine) power plants.

**RECENT WORK AND ACCOMPLISHMENTS** – Work on this project has been divided into two phases. Phase I, completed in June 1977, consisted of process development and detailed design of the pilot SRC processing skid. Phase II, currently in progress, includes fabrication and operation of the fuel processing skid as well as the completion of a combustor test program utilizing processed SRC as a fuel. Except for its high sodium (Na) and potassium (K) content, SRC has characteristics that appear to make it a viable candidate for gas turbine fuel. Its ash content, while being very low, is on the borderline (based on past experience with erosion problems on gas turbines burning pulverized coal) of being acceptable. Laboratory batch washing tests on SRC completed in Phase I, have resulted in significant reductions in both Na and K concentrations. The washing, carried out at temperatures and pressures exceeding 500°F and 700 psig, produces a fuel with low enough Na, K, and ash content to appear acceptable for gas turbine combustion.

**PLANS FOR THE COMING YEAR** – The pilot processing skid will be fabricated and operationally tested. Additional Phase II work includes design, fabrication, and operation on processed SRC fuel of a small-scale SRC combustion rig. Operational techniques will be developed for proper combustion startup and shutdown on SRC fuel, and parameters will be established relative to proper SRC atomization and combustion. Results of the combustion program should enable conclusions to be drawn regarding the potential suitability of SRC as a liquid boiler and gas turbine fuel. fuel.

#### COMMERCIAL-SCALE EXPANDED-BED HYDROPROCESSING OF SRC EXTRACT

CITIES SERVICE COMPANY

DOE - \$1,982,000

6/1/76 - 11/30/78

**OBJECTIVES** – This work is designed to demonstrate that SRC extract and expanded-bed hydroprocessing (LC-Fining) can be combined to produce a valuable liquid fuel or refinery feedstock from coal, and thus expand the commercial utilization of the SRC process. For this purpose, two tasks were established. Task I involves the determination of the optimum process conditions for hydrotreating SRC extract in an LC-Fining PDU. Task II included engineering studies and an on-site inspection of the Cities Service Lake Charles LC-Fining Unit, which would lead to the eventual processing of 10 to 15,000 barrels of SRC extract (Task III).

**RECENT WORK AND ACCOMPLISHMENTS** – PDU results showed that 60 to 65 percent of the SRC is converted to distillates in one pass through two expanded catalyst beds in series. A 30-day catalyst-aging run (one batch of catalyst) showed no appreciable catalyst aging at the above conversion level. Sufficient sulfur removal was achieved with a 90+ percent reduction in the sulfur contained in SRC converted to distillates and an 85 percent reduction in the sulfur contained in unconverted SRC. The nitrogen removal was 60 to 80 percent in SRC converted to distillates. The inspection of the Lake Charles commercial unit showed the high-pressure circuit to be in good mechanical condition. A 50 percent SRC/50 percent solvent blend can be handled in the steam tracing and pumping equipment available at Lake Charles.

**PLANS FOR THE COMING YEAR** – Work is being continued to reduce the nitrogen content in the SRC converted to distillates accompanied by an increased conversion of SRC to distillates. The convertibility of unconverted SRC bottoms will be determined by recycling them back to the expanded-bed reactor. The LC-Fining of SRC derived from Western coal and from low-severity extraction processing will also be investigated. Catalyst deactivation and makeup rate will be examined as a function of the ash content in the SRC.

#### EXXON DONOR SOLVENT COAL LIQUEFACTION PROCESS

EXXON RESEARCH AND ENGINEERING COMPANY

DOE - \$120,000,000; Carter Oil - \$75,000,000

Electric Power Research Institute - \$40,000,000; Phillips Petroleum - \$5,000,000

1/1/76 - 12/31/82

**OBJECTIVES** – Development of the Exxon Donor Solvent (EDS) coal liquefaction process, based on laboratory and engineering R&D efforts integrated with the operation of a 250-t/d coal liquefaction pilot plant, features development of technologies for coal liquefaction, solids separation, hydrogen and process fuel generation, and extensive product testing. The goal is to achieve com-

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mercial readiness in 1982 by obtaining all of the data needed for a commercial plant design. This objective will be accomplished by developing the EDS process and design bases necessary to liquefy a range of different coals in an environmentally acceptable manner, developing process correlations relating product yields and qualities to operating conditions and feed coals, translating program results into estimates of commercial plant economics and operations, identifying and implementing modifications to improve process economics and operability, developing and demonstrating process technology at the scale necessary to assure a reliable commercial design, and conducting an aggressive product test program to assure marketable products.

**RECENT WORK AND ACCOMPLISHMENTS** — Operation of the smaller integrated pilot plants has provided an increased understanding of the liquefaction of Wyoming coal. Increased liquefaction severity has proved beneficial in two respects: additional liquids are produced and a significant decrease in bottoms viscosity has been observed. Studies of the formation of  $\text{CaCO}_3$  agglomerates and reactor scale during liquefaction have yielded two methods of control. Pretreatment of the coal with sulfuric acid or sulfur dioxide has been very effective in the prevention of  $\text{CaCO}_3$  formation. Engineering economic analyses have shown both methods to cost less than 10 percent of cost of coal liquids, with the  $\text{SO}_2$  pretreatment cheaper by a factor of 5.

Operations of the 1-t/d coal liquefaction pilot plants have in part concentrated on key plant design issues for both the 250-t/d pilot plant and a commercial-size facility. Reactor feed slurry preheater studies showed minimum coking tendencies when the slurries were preheated in admixture with hydrogen. An additional study, conducted on the vacuum fractionation unit, showed minimal overhead entrainment and vacuum tower coking and confirmed the existing tower design for the 250-t/d pilot plant. Corrosion coupons were installed and preliminary analyses identified no major corrosion problems; additional detailed studies are underway to verify these conclusions. The large pilot plant and commercial liquefaction reactor designs have provisions for a solids withdrawal system to remove large, heavy slurry particles that would be atypical of the average particles in the feed slurry. Additional investigations have shown that solids withdrawal reduces  $\text{CaCO}_3$  found in the reactor by a factor of approximately 30 with Wyoming coal and has no effect on product yields. These results give confidence in the design for solids withdrawal in large pilot plants without affecting liquid yields.

Advances have been made in extending the integrated coking/gasification Flexicoking technology to coal liquefaction bottoms. Liquid yields for Wyoming coal bottoms have been shown to be 2 to 3 percent less on a coal basis than for Illinois coal bottoms. In separate experiments, gasification of coal-bottoms-derived fluid coke shows kinetics similar to petroleum-derived coke. In addition, these studies show that the fine particles ( $44\mu$  and smaller) produced during gasification have a very high ash selectivity. This high selectivity implies high carbon utilization in the gasification step. Studies on Flexicoking operability have been conducted in a 2-bbl/d integrated coking/gasification pilot plant to demonstrate Flexicoking operations, determine fine particle production rates and ash disposition, and provide additional data for scaleup needs. These studies show unit operability comparable to that in previous studies of petroleum residuum. The coke circulating between the coker and gasifier fluid beds contained over 40 percent ash and showed higher fine particle production rates than experienced on petroleum feeds, but this production was not excessive. Gasifier slagging is not considered a major concern, but additional studies for confirmation are underway.

Studies on process configuration and economics to provide a basis for understanding the commercial process as a whole have been conducted using a linear program process alternative model. The liquefaction, solvent hydrogenation, and fractionation section of the study design have been lumped together in the model depiction of a commercial study design along with separate depiction of bottoms processing, fuel production, and hydrogen generation processes. A number of other onsite and offsite processing blocks are also included in the model, such as coal preparation, steam and power generation, light ends recovery, sour water stripping facilities, ammonia recovery, general offsites, and others. The model considers simultaneously the many interactions and effects of alternate process bases, external price structures, and economic factors. Use of the model has provided rapid and consistent evaluations of laboratory results and process configurations within the framework of a commercial plant. Results from these studies have provided the bases for commercial study design updates for Illinois and Wyoming coals.

A series of combustion tests with Illinois coal liquids from the 1-t/d pilot plant were conducted using commercial equipment specially instrumented to facilitate measurement of critical temperatures, material balances, and fuel and exhaust compositions. Home-heating oil burner and industrial boiler tests were conducted using Illinois coal liquids as fuels and the data compared to results for ASTM No. 2 and No. 6 fuel oils. Smoke/particulates for the coal liquids tests were well within acceptable limits. Sulfur dioxide emissions were moderate and  $\text{NO}_x$  levels were high for raw coal liquids. Both were reduced when coal liquids hydrotreated to differing extents were used. The tests were conducted utilizing air/fuel ratio adjustments but without physical modifications to the combustion facilities, and they provide confidence in the potential of EDS products.

In construction activities for the 250-t/d pilot plant, subcontracts for engineering/procurement and construction have been awarded, design specifications have been completed, and detailed engineering design and procurement is underway. A cost control estimate and critical path schedule have been prepared to provide means for project control and implementation.

**PLANS FOR THE COMING YEAR** – Functional plans have been formulated to provide a means with which to gauge project accomplishments. These plans are to complete over 90 percent of the detailed engineering/procurement and approximately one-third of the construction for the 250-t/d pilot plant, expand the data base on Wyoming coal to allow selection of preferred EDS processing conditions, complete commercial study design updates for Illinois and Wyoming coals, complete process and engineering screening data for two additional coals, identify and implement additional process and engineering improvements, complete analysis of development requirements for EDS bottoms Flexicoking and update the development plan, and assess potential application of partial oxidation in EDS bottoms processing including economics and development requirements.

## **FLASH PYROLYSIS COAL LIQUEFACTION PROCESS DEVELOPMENT**

**OCCIDENTAL RESEARCH CORPORATION**

**DOE - \$3,780,000**

**6/28/76 - 6/30/78**

**OBJECTIVES**– The purpose of this program is to test ORC's Flash Pyrolysis process for production of liquid fuels from coal in a 3-t/d process development unit. In this process, a single-stage reactor is used to conduct short-residence-time pyrolysis of bituminous (caking) coal without oxidative pre-treatment. The char co-product can be desulfurized and directly substituted for the parent coal as a



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utility fuel. This approach is an attractive alternative to total conversion via hydroliquefaction because of its intrinsically higher thermal efficiency, which adds to its potential economic advantage. Specific objectives include demonstration of reactor operability for extended periods, PDU confirmation of high liquid yield, product evaluation, and an assessment of the commercial viability of the process.

**RECENT WORK AND ACCOMPLISHMENTS** — Three subbituminous coal pyrolysis baseline runs were completed. The objectives of making three runs of 1-week duration and obtaining reproducible baseline material balance data were achieved. Operability of the unit was thus demonstrated. The pyrolysis reactor has been operated with bituminous (caking) coal without fouling, which confirms the technical feasibility of this concept. "Flash Pyrolysis" tar from the baseline runs was hydro-refined using commercially available petroleum catalysts. High conversion of preasphaltenes and asphaltenes to oils and high sulfur removal are obtainable under conditions of moderate severity.

**PLANS FOR THE COMING YEAR** — Extended runs processing bituminous coal will be conducted to obtain detailed material balance data and products for evaluation.

#### ADVANCED COAL LIQUEFACTION COMMERCIAL PLANT

FLUOR ENGINEERS AND CONSTRUCTORS, INC.

DOE - \$2,701,024

4/21/76 - Continuing

**OBJECTIVES** — This effort was undertaken to design a commercial facility to produce a product slate comprising principally liquid synthetic fuels by an advanced coal liquefaction scheme and to appraise the economic viability of this facility. The goals were threefold; namely, economic, technical, and demonstration plant recommendations. It was expected that the results of this study could focus on the maximum price U.S. energy industries would be willing to pay for imported oil and provide a viable alternative to petroleum as a gasoline source. It would also contribute significantly to employment and economic growth in the plant geographic areas.

**RECENT WORK AND ACCOMPLISHMENTS** — The draft of the final report has been completed and, after review, will be ready for publication. This report presents the results of an in-depth engineering design and economic analysis of the commercial-scale production of gasoline from high volatile bituminous type A (Pittsburgh No. 8 seam) eastern coal via an advanced coal liquefaction process. The plant processes 30,000 t/d (moisture free basis) of cleaned coal from a captive underground mine to produce 66,000 bbl/d of unleaded regular grade gasoline and 77.5 MMscf/d of SNG plus by-products of propane, butane, ammonia, sulfur, and phenols. The overall configuration is a grass roots, totally self-sufficient facility. The total capital requirement (mid-1977) is \$2.6 billion. The transfer cost of coal from the mine is \$25.10/ton. A gasoline selling price of \$24.35/bbl (\$4.30/MMBtu of total plant products) using a pretax ROI of 14 percent, results from the economic analysis. The reported economic analysis includes sensitivity studies of the effect on product price of variations in capital cost, rate of return, debt/equity ratio, coal cost, debt interest rate, and by-product SNG selling price. Additional data and development requirements have been defined, and, if successfully derived, could bring gasoline selling price for the design coal to less than \$19/bbl.



The core liquefaction technology employed is hydrogen donor solvent extraction with relatively standard petroleum refining techniques used for production of finished products. Analyses of the technology indicate a high degree of inherent flexibility to adapt to alternative design bases and objectives such as other coals and product slates. Minor design modifications in the liquefaction area are readily effected to adapt the process to accept a wide range of lower rank eastern coals such as Illinois No. 6 and even western coals such as Kaiparowitz and Navajo Reservation. With more extensive design modifications, North Dakota lignite and Montana Rosebud can be satisfactorily processed. Revisions to the process can also be readily made to produce an alternative product slate such as boiler fuel, gas turbine fuel, jet fuel, diesel fuel, or synthetic crude oil. Assessment of the basic technology has established that there are no areas of uncertainty critical to the eventual technical success of the project. Where minor design uncertainties may exist, it would be limited to impacting only on yield or throughput and can be compensated for with extra design margins. The soundness of the basic technology and flexibility of the processing scheme coupled with the attractive economics make the advanced coal liquefaction process an excellent candidate for consideration to move from pilot plant scale to the demonstration or pioneer plant level.

**PLANS FOR THE COMING YEAR** – The final interim report will be published in the first calendar quarter of 1978. Work will proceed on three additional areas of the conceptual design, namely, (1) trade-off studies on refining sequences, (2) evaluation of internal power generation versus purchased power, with fuel gas methanation for SNG sale, and (3) plant study configuration for maximum syncrude production.

#### COAL-TO-METHANOL-TO-GASOLINE PLANT DESIGN

BADGER PLANTS, INC.  
DOE - \$2,585,610  
7/76 - 3/79

**OBJECTIVES** – This effort was undertaken (1) to prepare a conceptual design package of a coal-to-methanol commercial plant based on a nominal 50,000 t/sd coal feedrate on a moisture and ash free basis. The conceptual design package is to include a capital cost estimate and economic evaluation of the grass roots commercial plant based on a standard rate of return over a 20-year life of the plant. Recommendations will be made for a demonstration plant which could be one train of the commercial facility to produce principally methanol and methanol fuel. Also a conceptual design package of a methanol-to-gasoline commercial plant utilizing the Mobil M-Process will be prepared. This conceptual design is to be integrated with the coal-to-methanol conceptual design to produce principally high-octane gasoline. By-products could include LPG and high-Btu gas. Resultant costs and economics of the coal-to-methanol-to-gasoline facility are to be compared with other processes producing gasoline and liquid fuels from coal. As the developing shortage of oil and gas becomes more acute, it will force a reassessment of energy economics. This reassessment should indicate that the commercial production of large quantities of clean synthetic fuels, such as methanol and gasoline from coal, is feasible within the coming decade.

**RECENT WORK AND ACCOMPLISHMENTS** – A draft of the interim final report is being completed covering the conceptual design package for the coal-to-methanol commercial facility. The completed work will include a capital cost estimate and economic evaluation which should indicate that the commercial production of methanol from coal is feasible. The conceptual design

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study is based on a slag bath type gasifier as specified under the terms of the contract. This work led to an alternate design incorporating a continuous drain slagging type gasifier, which may be preferred. Work is proceeding toward the development of a conceptual design package of the methanol-to-gasoline facility based on the Mobile M-Process for conversion of methanol to gasoline.

**PLANS FOR THE COMING YEAR** — Approved copy of the interim final report for the coal-to-methanol portion will be issued to DOE on February 15, 1978. Process flow diagrams and equipment specifications will be developed for the methanol-to-coal conceptual design study. Capital cost estimates and economic evaluations will be completed.

## **PRELIMINARY DESIGN SERVICES**

**RALPH M. PARSONS COMPANY**

DOE - \$3,280,154

12/31/74 - 4/30/78

**OBJECTIVES** — This project is designed to assist DOE/Fossil Energy in development of viable commercial coal conversion plants by preparing multiple conceptional designs that preview design/operating characteristics and projected economics of commercial-scale multi-product coal conversion complexes. Captive coal mines and power plants will be included. The best available data, from any source, will be used, and additional data plus equipment requirements that will assure reliable performance of the plants will be defined. The primary impact of this work is to display a cohesive definition of the probable characteristics, potential performance, and potential economics of large future coal conversion complexes. For the high potential configuration designs, a cost-benefit basis is provided for establishing priorities for process and equipment development programs.

**RECENT WORK AND ACCOMPLISHMENTS** — Three conceptual designs were completed: Fischer-Tropsch, Oil/Gas, and Pogo. R&D reports describing the designs and economic evaluations for two of these, Fischer-Tropsch and Oil/Gas, were published. A paper describing the Pogo design was prepared for presentation to a national American Institute of Chemical Engineers meeting in November 1977. Approximately 75 percent of the preliminary design concept definition was completed for the Multi-Process Demonstration Plant and conceptual design/economic evaluation has begun for three Large Prestressed Concrete Vessels for projected use in coal gasification and liquefaction plants. Four major R&D reports and 16 additional summaries were published in the field of coal conversion. Included was a description of procedures to produce petrochemicals from coal. These preliminary conceptual designs are summarized here.

The objective of the Fischer-Tropsch conceptual design was to define desirable characteristics and projected potential for large second generation Fischer-Tropsch plants to be responsive to U.S. requirements. The design includes a captive coal mine to produce 40,000 t/d of run-of-mine coal in the eastern region of the U.S. Interior Coal province. After cleaning and preparation, 30,000 t/d of coal is converted to about 260 MMscf/d of substitute natural gas (SNG) and 50,000 barrels per day of liquids. The fuel products are premium quality, having no sulfur, nitrogen and particulate matter. Using flame sprayed catalytic heat exchanger reactors, the thermal efficiency, coal to fuel products, is projected to be about 70 percent. The complex doesn't require a power plant for normal operation; energy required to generate the electrical power and steam for operation of the complex is recovered from process generated heat. A number of process and equipment developments must

be successfully completed to convert the design to reality; these developments are described in a report prepared for DOE. Projected required average product selling prices, fourth quarter 1976 dollars are as follows: at 100 percent equity \$3.30 per million Btu; and at 65/35 ratio of debt/equity, \$2.25 per million Btu. Financial parameters include a 5-year design-construction period, 9 percent interest rate, and a 20-year operating life. The Oil/Gas conceptual design uses SRC II technology. The captive coal mine would produce 47,000 t/d of run-of-mine coal in the eastern region of the Interior Coal Province. The process plant would convert 36,000 t/d of clean washed coal to about 165 million scf/d of SNG and 75,000 bbl/d of liquid fuels. The projected thermal efficiency, coal to fuel products, is of the order of 75 percent. The conceptual design report defines process and equipment developments required to convert the design to reality. The projected required product selling prices using fourth quarter 1976 dollars and project parameters equal to those described earlier for the Fischer-Tropsch design are as follows: at 100 percent equity, \$2.50 per million Btu; and at 65/35 ratio of debt/equity, \$1.95 per million Btu.

The Pogo Conceptual design represents a coal refinery with a captive coal mine; Pogo is a DOE acronym for power-oil-gas-other. Phase I of the design project was completed and the R&D report submitted; this included assessment of the technical and economic factors for candidates from each generic class of coal liquefaction process and a recommended basis for the final design. The base case design for Pogo was completed; it was conceived to be located in the eastern region of the Interior Coal Province. Preliminary assessments of designs for two other locations will be developed. The base case design converts approximately 45,000 t/d of clean coal to about 150 million scf/d of SNG, 15,000 bbl/d, 35,000 bbl/d of pool gasoline, 27,000 bbl/d of distillate fuel oil, 1,600 t/d of a premium grade coke, and 1,000 Mw of electrical power for sale. The power plant design defined a high efficiency combined cycle configuration which interfaced with the coal conversion plant. The projected efficiency of conversion of captively produced fuel gas to electrical power is about 44 percent. The conceptual Multi-Process Demonstration Plant is conceived to contain a low-pressure fuel gas gasifier, several intermediate-pressure synthesis gas gasifiers, a liquefaction section, and a combined cycle power plant. The design was in a late stage of completion at the end of the fiscal year. Conceptual design for converting Petrochemical Feedstocks and Chemicals from coal included development of comprehensive processing facilities to convert coal to petrochemical feedstocks, petrochemicals, and chemicals. The design concept uses Fischer-Tropsch and Oil/Gas coal conversion techniques to produce ethylene plus a number of aromatic and aliphatic products. The conceptual design and economic evaluation of three Large Prestressed Concrete Vessels (up to 25 feet diameter and 2000 psig) constructed of prestressed concrete was begun. These vessels have the potential advantage of providing large economic vessels for use in coal gasification and liquefaction plants without the limitation of availability of shop and field fabrication facilities required to roll and weld the thick sections required for metal vessels. The technical practicality and projected economics of the prestressed concrete vessels will be compared with metal vessels.

**PLANS FOR THE COMING YEAR** — The Pogo alternate case designs and economic projections will be completed, and published in a report. Work on the Multi-Process Demonstration Design and three preliminary designs of large Prestressed Concrete Pressure Vessels will be completed and reports describing the results will be issued. This contract is scheduled to be completed during FY 1978.

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## FLUID COKING OF COAL LIQUEFACTION RESIDUES

EXXON RESEARCH AND ENGINEERING COMPANY

DOE - \$1,312,470

7/1/76 - 11/1/78

**OBJECTIVES** – This program is to evaluate the applicability of commercial fluid-bed coking technology to processing the residues from coal liquefaction processes. Solids will be separated from primary coal liquids by methods such as hydroclones, vacuum distillation, and solvent deashing. The residues from these separation processes will contain both solids and valuable liquid products. The fluid coking process has the potential of recovering these liquids from coal liquefaction residues. The work in this program includes analytical and bench-scale testing of three residues with the most promising being tested in a nominal 2-bbl/d fluid-bed unit.

**RECENT WORK AND ACCOMPLISHMENTS** – The first residue evaluated was a sample of H-Coal solvent precipitation underflow. Sample characteristics caused operational problems, which led to program modification. These sample characteristics rendered questionable the representativeness of the sample and the applicability of the resultant data to the liquefaction process scheme from which this sample was generated. Data obtained with this sample indicated that fluid-bed coking of the residue could recover heavy liquids, and operability would not be limited by bogging in the temperature range of interest. Stripped SRC antisolvent deashing underflow is the second of three candidate coal liquefaction residues to be evaluated. Analytical characterization and bench-screening studies of the coke, liquid, and gas yields have been completed as well. A simulated coker liquid recycle run was completed in the small stirred coker using coker liquid generated in the once-through runs as feed. The results from this run will be used to evaluate the yield impact of operating the coker in a recycle mode. This residue contains a substantial amount of material boiling below 1000°F. Both the bench and stirred-coker results indicate that all of this material can be recovered along with additional higher boiling material using the fluid coking process. As has been observed for H-Coal solvent precipitation underflow and EDS coal liquefaction vacuum bottoms, minimal conversion of heavy 1000°F<sup>+</sup> to 1000°F<sup>-</sup> boiling material occurs. This observation was supported by the results from the simulated recycle run, where no conversion to 1000°F<sup>-</sup> boiling material was observed.

**PLANS FOR THE COMING YEAR** – The latest small stirred-coker runs will be element balanced and the results analyzed including an evaluation of the yield impact of recycle coking. Inspection properties of the coker liquids produced from the stripped SRC antisolvent deashing underflow will be determined as a measure of product quality. An assessment of the operability of fluid-bed coking with this feed will be made in a small fluidized-bed unit. One additional residue, H-Coal vacuum bottoms, is scheduled for evaluation during 1978. The most promising of the three candidates will then be processed in a 2-bbl/d unit, which will provide an assessment of the operability of fluid coking in an integrated two-vessel system.

## CLEAN COKE PROCESS

USS ENGINEERS AND CONSULTANTS, INC.  
DOE - \$8,337,176; U.S. Steel - \$3,455,153  
3/72 - 8/78

**OBJECTIVES** – The clean coke process uses both fluid-bed carbonization and hydrogenation/liquefaction to convert high-sulfur coals to low-sulfur metallurgical coke, chemical feedstocks, and liquid and gaseous fuels. This project includes demonstration of process feasibility at the PDU level, initially with Illinois No. 6 coal and subsequently with Western Kentucky and Pittsburgh seam coals, and refinement of a process engineering design for a large commercial plant. A principal goal for the current year was completion of all PDU studies with Illinois coal, including liquefaction and cokemaking; another was completion of carbonization studies with Kentucky coal. The clean coke process shows promise for providing the steel industry with a way to use high-sulfur coal to produce high-quality metallurgical coke. Moreover, both the carbonization char and hydrogenation heavy oil are low-sulfur, high-Btu fuels, well suited for power plant or industrial use.

**RECENT WORK AND ACCOMPLISHMENTS** – Parametric studies and process-development work, including material balances, were completed for the fluid-bed carbonization of Kentucky No. 9 seam coal in the PDU. Coal containing 4.0 percent sulfur was processed to char containing less than 0.5 percent sulfur. Complementary to this program was an extensive metals-corrosion study in the carbonization atmosphere. The study was completed and will contribute substantively to the selection of optimum materials of construction for large-scale carbonization units. Parametric studies and process-development operations were also completed with Illinois No. 6 seam coal in the carbonization PDU. Modifications in equipment and method of operation resulted in significant simplification of both liquefaction and vapor-stripping operations. In liquefaction, use of run-of-mine coal as feed resulted in MAF coal conversions as high as 93 percent and eliminated the need for catalytic rehydrogenation of the slurry oil before recycling. These improvements are attributed to a beneficial catalytic action of the ash in run-of-mine coal. In vapor stripping, improvements in heaters and the letdown valve enabled recovery of as much as 95 percent of the oil from the converter in a single stage and eliminated the need for second-stage stripping. The low-sulfur char was pelletized with hydrogenation-derived binder (cutback SRC), and the pellets were processed to satisfactory metallurgical coke in the cokemaking PDU.

**PLANS FOR THE COMING YEAR** – Process-development work in the PDUs will be completed, including fluid-bed carbonization of Pittsburgh coal.

## PROJECT LIGNITE

UNIVERSITY OF NORTH DAKOTA  
DOE - \$5,116,000  
3/72 - 6/78

**OBJECTIVES** – Originally, this work was to identify or develop the technology for the conversion of lignite to a spectrum of upgraded fuel products and chemical intermediates by a combination of drying, carbonization, gasification, liquefaction, and hydrogenation, to permit flexibility of product output and optimization of process economy. North Dakota, Montana, and Wyoming have resources of 440 billion tons of lignite that have been exploited only minimally. Starting in June 1977, a 1-year extension was granted to obtain adequate data on which to base a decision regarding

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scaleup of the Solvent Refined Lignite (SRL) process at an existing facility, and also to provide data for new economic evaluations of the process.

**RECENT WORK AND ACCOMPLISHMENTS** — Further studies of operating variables in the 0.6-t/d PDU for the manufacture of SRL have resulted in the definition of conditions that are optimal from both the process standpoint and the mechanical operability standpoint. The operability of the liquefaction section has been improved by modifications to the vacuum bottoms draw-off system of the vacuum flash separator. Process solvent recovery for recycle has been improved by modifications in the flow to provide for fractional distillation of the oil fraction from the oil-water separators to produce additional recycle solvent as the fractionator bottoms. The optimum pressure for the liquefaction reaction is 2500 psig, which is the maximum practical operating pressure for the reactors. Lower pressures result in decreased oil and SRL yields and lower recycle solvent recoveries at comparable conversion levels. Operable charge rates are superficial liquid hourly space velocities around 1.4 and superficial gaseous space velocities around 400 scf/hr per ft<sup>3</sup> of reactor volume. The minimum practical solvent-to-coal ratio appears to be 1.6 to 1.8; at low ratios the vacuum bottoms product is too viscous or high melting to handle and at high ratios recycle solvent recovery decreases. Under these conditions the optimum dissolver outlet temperature is about 825°F (440°C); conversion, as measured by the pyridine solubles in the vacuum bottoms decreases both above and below this temperature. The optimum hydrogen-to-carbon-monoxide ratio in the feed gas appears to range from about 1.0 to about 1.5 (50 to 60 percent hydrogen when only syngas is charged). Above and below this range, gas yields increase and oil and SRL yields decrease. Two extended runs have demonstrated that operation with total recycle of solvent (no makeup solvent added) did not adversely affect conversions and yields, even when continued until the solvent appeared to be lined out, and that the recycle of the bulk of the product gas after caustic scrubbing to remove carbon dioxide and hydrogen sulfide reduces the makeup gas requirements appreciably and appears to have no adverse effect on conversions, yields, or operability. The solid-liquid separation system even after modification has not been proved operable except for very short periods.

**PLANS FOR THE COMING YEAR** — Another 28-day run will be conducted, and further studies of various lignites will be made. Operations with only the preheater in the reaction circuit will be attempted to determine how much conversion occurs in 10 to 20 seconds in the preheater, both at normal and at higher temperatures, as compared with the conversion occurring when a reactor with 30 to 45 minutes residence time follows the preheater. Both as-received and dried lignite will be liquefied using only pure hydrogen as the charge gas to compare lignite with bituminous coal used in this type of operation elsewhere. Special attention will be given to the solid-liquid separation system to achieve proper operation and production of test quantities of deashed SRL. In the liquefaction reactor, buildup of loose solids of such a particle size that they are not carried will also be investigated. Corrosion test coupons were placed in the reactor at the start of the 28-day runs, and will be examined at the completion of the three runs. Future efforts should address the problem of possible long-term buildup of inorganic solids in the reactors and the possibility of improving the hydrogen donor property of the solvent.

## CO-STEAM PROCESS

GRAND FORKS ENERGY RESEARCH CENTER

DOE - \$500,000

10/1/76 - Continuing

**OBJECTIVES** – The goal is to develop an economic second-generation process for liquefaction of low-rank coals by reaction with steam and synthesis gas (a mixture of hydrogen and carbon monoxide). The near-term objective is to demonstrate that modestly higher reaction temperature and a hydrogenated recycle solvent can be used to: balance the consumption of  $H_2$  and CO from synthesis gas, reduce residence time in the reactor to 10 to 20 minutes, permit some reduction in pressure below 4000 psi, and improve the viscosity of the product while maintaining yield.

**RECENT WORK AND ACCOMPLISHMENTS** – Work at GFERC includes: determination of reaction kinetics using a hot-charged and time-sampled batch system, process and reactor optimization using a 5-lb coal/hr continuous processing unit, and analytical facilities dedicated to the project. Studies completed in the batch reactor have demonstrated that carbon monoxide reacts rapidly with lignite at all temperatures from 440° to 480°C, with part of the CO combining with oxygen in the coal and the remainder reacting more slowly with water to supply in-situ hydrogen. Hydrogen is consumed far more slowly than carbon monoxide from synthesis gas. The quality of the product measured by the molecular weight distribution of the distillation residue was greatly affected by the combination of temperature and the hydrogen donor activity of the solvent. With suitable donor constituents present, which could be provided by hydrotreating the recycle solvent in a continuous process, a high-quality product was produced in residence times under 20 minutes and at temperatures above 460°C; and, repolymerization and coking were essentially avoided and loss of yield to methane production was acceptable. Major emphasis on analytical methods has been to improve procedures for characterizing coal-derived liquids using GC-MS, LVMS, column chromatography, and HPLC employing calibrated gel permeation columns, and to determine the chemical and physical properties of product fractions that contribute to high viscosity.

**PLANS FOR THE COMING YEAR** – The continuous unit constructed this year is undergoing shakedown and will be used to demonstrate feasibility of a high-temperature and short-residence time process based on use of a hydrogenated recycle solvent. Followup work will involve systematic study of residence time distribution and level of turbulence, using different reactor modules and recycle of gas and liquid to improve reactor design. Pressure reduction will be studied by lowering the vapor pressure of the solvent and partially drying the coal under conditions that avoid deactivation. A more detailed study will be made of process economics to determine the cost savings potential of the technical results. In the batch system, work will continue on improvement of recycle solvent properties to reduce repolymerization. Further studies will be conducted on factors affecting the yield of the volatile oil fraction. Studies on effects of coal feed will include particle size, methods of drying, and differences in coal mineral content. Known additives will be investigated to determine their effect on product quality. Fate of trace elements will be studied using the analytical capabilities of the new inductively coupled argon plasma spectrometer, and development will be initiated to apply the field desorption source for the mass spectrometer to analysis of heavy product fractions.



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## FILTRATION PROCESS AND EQUIPMENT STUDIES

JOHNS-MANVILLE SALES CORPORATION

DOE - \$366,963

5/22/75 - 6/22/78

**OBJECTIVES** – One aim of this program is to demonstrate solids-liquid separations for SRC at filtration rates approaching theoretical performance. Efficiencies of solids separation from liquefied coals on a rotary pressure precoat filter were demonstrated. A 0.1-ft<sup>2</sup> test leaf capable of operating at high temperatures and pressures was tested. A second goal is to predict optimum startup conditions for a 50-ft<sup>2</sup> pilot rotary pressure precoat filter being installed at the Fort Lewis SRC pilot plant in 1978. Separation of ash and sulphur from liquefied coals by filter-aid filtration is: positive (solids cannot “breakthrough” into the product) and quickly adjustable to make changes in process conditions. It also results in minimum product losses and produces a “dry” filter cake with minimum product and entrained solvent content. Such filtration will enable the production of SRC of sufficiently low ash and sulphur to meet EPA emission standards and is also applicable to other liquefied coal processes where an efficient method of solids removal is required.

**RECENT WORK AND ACCOMPLISHMENTS** – Samples of filter feed from various coals from pilot plants at Wilsonville and Fort Lewis were tested for filtration properties. The effects of grade of precoat material, blade advance rates, differential pressure, temperature, and drum speed on filtration efficiency were studied. Filtration rates for one coal, which was poorly converted, in excess of 750 lb/ft<sup>2</sup> of filter area/hr were obtained. Other liquefied coals gave rates of 500 to 600 lb/ft<sup>2</sup> of filter area/hr. Filter-aid (precoat) usage of 4 to 7 percent by weight of insolubles was obtained. Filtration rates of day-old samples of Fort Lewis filter feed were compared with those filter feeds that had been allowed to cool and stand for several months. These tests showed the “fresh” sample to have similar rates, but slightly finer particle-sized solids. Installation of a device to permit precoating the test leaf at high temperatures and pressures began in September.

**PLANS FOR THE COMING YEAR** – The in-situ precoat device will be installed. Optimization studies will continue on the 50-ft<sup>2</sup> pilot rotary precoat filter. The effects of high-temperature high-pressure precoating on the filtration operation and precoat properties will be studied. Tests will also be run on filter feeds made from other liquefied coals.

## RECOVERY OF OILS AND GASES BY PYROLYSIS

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$1,067,000

1976 - Continuing

**OBJECTIVES** – The goals of this program are to design and develop pyrolytic processes for recovering oils from the residues produced by coal liquefaction processes and to determine the effect of these pyrolytic processes on the overall economics of the coal liquefaction processes.

**RECENT WORK AND ACCOMPLISHMENTS** – A Fluidized-Bed Solvent Recovery System has been installed in a newly constructed building. All piping connections and modifications have been completed, and instrumentation and control circuitry are being installed. The rotary calciner was relocated adjacent to the Fluidized-Bed Solvent Recovery Facility so that a single-feed system will supply both test units, and solutions to operating problems in feeding primary separation residue



will benefit both units. Preliminary tests with the rotary calciner indicate that the addition of char to the primary separation residue greatly reduces agglomeration with the calciner; therefore, a dual-feed system for feeding hot recycle char and primary separation residue simultaneously to the calciner was developed. Initial tests of this system indicate that the agglomeration problem can be eliminated. A cold model of the Fluidized-Bed Solvent Recovery System was constructed and is being utilized to determine residence times in the draft tube and main bed as a function of space velocities and feed rates.

**PLANS FOR THE COMING YEAR** — Both the rotary calciner and the fluidized-bed systems will be operated to determine optimum operating variables for maximum recovery to make oil from the primary separation residue. The cold model study will be completed, and the information will be used in developing a mathematical model of the fast fluidized-bed system.

#### **CRESAP TEST FACILITY**

**FLUOR ENGINEERS & CONSTRUCTORS, INC.**

DOE - \$51,998,595; American Electric Power Service Corp. - \$1,000,000

Allegheny Power Service Corp. - \$1,000,000; Fluor - \$791,880

5/14/74 - 2/15/78

**OBJECTIVES** — The reactivation, renovation, and conversion of the Cresap Pilot Plant for production of low-sulfur fuel oil is the initial goal of this program. Then the plant will also be provided with the capability for component and equipment testing. Included will be the development and/or testing of equipment components useful in any or all processes for converting coal into a clean liquid fuel suitable for electric power generation and other industries. Following the renovation, there will be a period of extended operation to demonstrate an integrated coal liquefaction process to achieve coal liquefaction and desulfurization.

**RECENT WORK AND ACCOMPLISHMENTS** — Construction activities at the Cresap Test Facility have been completed. All of the offsites and utilities are operational. All process units with the exception of the hydrogenation section have been operated on petroleum-derived liquids, followed by operation on coal and startup solvent to produce granulated coal-derived extract. Startup activities leading to operational status of the high-pressure hydrogenation section are continuing.

**PLANS FOR THE COMING YEAR** — Work will include some additional operation of the front-end units to produce a granulated extract. Also, startup activities will soon be completed on the hydrogenation units, which will be operationally integrated into the plant to achieve the objective of demonstrating an integrated coal liquefaction process that will produce a low-sulfur low-ash content boiler fuel oil.

#### **CHEMICALS FROM COAL**

**THE DOW CHEMICAL COMPANY**

DOE - \$386,808; Dow - \$34,104

4/16/74 - 9/1/77

**OBJECTIVES** — This contract was to define a process and make preliminary capital and operating cost estimates for a commercial-size plant to produce petrochemicals from coal liquefaction products. Process studies were made on liquids from four coal liquefaction processes—H-Coal,

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SRC-II, COED, and Synthoil. These evaluations were used to define the suitability of the four samples as chemical feedstocks. Based on comparisons of process study results, one of the four coal liquefaction processes was selected for further study. This study included material balances and utility requirements for each major process step, a preliminary process flowsheet, and estimates of capital and operating costs.

**RECENT WORK AND ACCOMPLISHMENTS** – Experimental work was completed on three liquids (H-Coal, COED, Synthoil) prior to this year. Work on SRC-II was started earlier but completed during this reporting period with the reforming of the two-pass hydrotreated SRC-II straight-run naphtha. Interim reports present the results of an investigation to determine the suitability of the COED, H-Coal, Synthoil, and SRC-II syncrudes as petrochemical feedstocks. Based on the results of laboratory investigations, conversion of the liquid products to petrochemical feedstocks per ton of coal fed to each liquefaction process was 15.0, 33.9, 26.9, and 23.0 percent, respectively. While it is recognized that the samples obtained were from liquefaction runs that were not necessarily representative, based on the samples that were furnished the H-Coal process produces the maximum yield of feedstocks and thus was chosen as the basis for preliminary design and economics for a commercial chemical refinery. From a chemical refinery sized to process 50,000 bbl per stream day of H-Coal derived syncrude, yields of 653,000 lb/year of ethylene and 245,000 gal/year of benzene were calculated. Total annual revenues were estimated, and total capital investment was also calculated. The results of this investigation demonstrate both the technical feasibility and economic viability of processing coal-derived syncrudes for future production of petrochemical feedstocks.

**PLANS FOR THE COMING YEAR** – This contract was completed during the year.

## CONVERSION OF METHANOL TO HIGH-OCTANE GASOLINE

MOBIL RESEARCH AND DEVELOPMENT CORPORATION

DOE - \$673,643; Mobil - \$224,548

9/28/76 - 5/31/78

**OBJECTIVES** – The goal is to develop a fluid-bed process for the conversion of methanol to high-octane gasoline using Mobil's proprietary catalysts. The scope of work includes design, construction, and operation of a 4-bbl/d fluid-bed pilot plant. Specific objectives are to: quantify the effects of major process variables on conversion and product yields, demonstrate steady-state operation with desired gasoline yield, define catalyst stability, evaluate selected properties of the product gasoline, and develop scaleup information for a larger unit. Coupling of this process with the commercially established technology for the conversion of coal to methanol provides a route to obtain high-octane gasoline from coal. This process provides better product selectivity and thermal efficiency with lower capital investment than the Fischer-Tropsch process, which is the only one in commercial use for the conversion of coal to gasoline. Based on progress to date, this process could make the conversion of coal to high-octane gasoline available for commercialization at a relatively early date.

**RECENT WORK AND ACCOMPLISHMENTS** – Construction of the 4-bbl/d pilot plant was completed on schedule. The original design was improved significantly from the results of flow studies conducted in a full-size glass model of the reactor. Gas velocity effects on catalyst entrainment rate, bed density, and reactor inventory were determined and used in the design calculations.

The pilot plant has a 4-in-I.D. by 25-ft-high dense fluid-bed reactor with a 2½-in external line for catalyst recirculation. A 10-in-I.D. by 32-in high regenerator is integrated with the reactor. Pilot plant operation with methanol was started in September 1977. The startup was exceptionally smooth, and an initial run of 33 days was achieved without any difficulties. Complete methanol conversion was obtained at design conditions, and the C<sub>4</sub> + gasoline yield was 88 wt percent (of total hydrocarbons) or higher. The reactor temperature profile was uniform over most of the reactor. Product selectivity and gasoline quality were comparable to previous results from bench-scale units. The pilot plant was then shut down for some mechanical modifications including installation of improved feed distributor and cyclone.

**PLANS FOR THE COMING YEAR** – The unit will be restarted to study the major process variable effects on product yields. Catalyst stability and regenerability will be studied further. This run will be continued for about 3 months, with results used to quantify the effects of process variables on product yields and pilot plant operation. Some selected modes of operation such as liquid/vapor feed and light gas recycle will be studied. Experimental work on the pilot plant is tentatively scheduled for completion in January 1978. The final report will then be prepared.

## UPGRADING OF COAL LIQUIDS

UOP INC.  
DOE - \$997,782  
2/1/77 - 1/30/79

**OBJECTIVES** – This program is determining the processability of coal distillates by conventional petroleum refinery practices. The work is being done in continuous bench-scale equipment, using commercial catalysts and process conditions. Specific objectives are to process selected DOE-sponsored coal liquid distillates through a comprehensive hydrocracking scheme; determine the response of hydrotreated coal liquid distillates to fluid catalytic cracking; maximize the production of either motor gasoline or No. 2 fuel oil by hydrocracking; establish yield-octane relationships for the catalytic reforming of naphthas produced in primary coal liquefaction programs and/or by hydrocracking coal-derived gas oils; and correlate feedstock properties and process conditions data with product yields and qualities. Samples of naphtha and gas oil from both the Exxon Donor Solvent (EDS) and H-Coal processes have been furnished for carrying out the program.

**RECENT WORK AND ACCOMPLISHMENTS** – Primary coal naphthas from both the EDS and H-Coal processes were successfully hydrotreated to reduce nitrogen, sulfur, and oxygen contents to levels tolerable in subsequent catalytic reforming. Process conditions required were relatively more severe than for petroleum naphthas, which is consistent with the high heteroatom content of these naphthas. The hydrotreated products contained a large proportion of aromatic and naphthenic hydrocarbons. Accordingly, the subsequent catalytic reforming required relatively mild conditions to give high yields of 100-octane gasoline. The H-Coal gas oil was processed over a first-stage hydrocracking catalyst using a range of pressures, temperatures, and space velocities. These tests provided data for subsequent correlation of degree of upgrading with process conditions. Based on this process variable study, preparative runs were made to provide upgraded charge stocks for second-stage catalytic hydrocracking to distillate fuel oil and gasoline. In the fuel oil operation, the second-stage effluent was continuously fractionated to give a 600°F (416°C) end-point overhead product, while the bottoms were recycled. Temperature was adjusted to a level where there was no net yield of bottoms material. Likewise, in the gasoline operation, the effluent was continuously

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fractionated to give a 375°F (191°C) overhead product, and the bottoms were recycled to extinction. The gasoline operation was carried out over a range of pressures and space velocities. Hydrocracked naphthas, produced at two pressure levels, were catalytically reformed. As in the case of hydrotreated primary naphtha, a high yield of 100-octane gasoline was obtained at relatively mild conditions.

**PLANS FOR THE COMING YEAR** — First- and second-stage catalytic hydrocracking studies will be extended to the EDS process gas oil. Both the EDS process gas oil and the H-Coal gas oil will be hydrotreated and processed in a small-scale fluid catalytic cracker. Data from the several process variables studies will be correlated, and final yield structure from the various processes will be calculated.

## HYDROCARBONIZATION RESEARCH

OAK RIDGE NATIONAL LABORATORY  
DOE - \$900,000  
10/1/74 - Continuing

**OBJECTIVES** — This work is designed to estimate optimum conditions for operation of fluidized-bed hydrocarbonization reactors; operate a fluidized-bed reactor capable of processing 10-lb/hr of coal at conditions up to and including 1200°F and 20 atm; and to establish hydrocarbonization conditions favoring operability, low-char yield, and attractive economics. Hydrocarbonization is an important type of coal conversion process that combines moderate temperatures and pressures with fluidized-bed operation using hydrogen-rich gas for fluidization. It produces hydrocarbon liquids, gaseous fuels, and solid char. Based on the coal used and the operating conditions, the yields may be varied both in quantity and quality. This bench-scale investigation of hydrocarbonization will provide yield data for optimizing various processes and contributions to technology areas such as handling of caking coals in fluidized-bed reactors.

**RECENT WORK AND ACCOMPLISHMENTS** — More than a dozen tests have been completed successfully using Wyodak Western subbituminous coal. Based on these experiments, it has been concluded that useful products could be extracted at modest prices by mild hydrocarbonization of Western coal. Such a process has the following attributes: significant quantities of oil and gas can be produced without the need for a gasifier and oxygen plant to produce hydrogen; the residual char is an efficient, low-sulfur boiler fuel that does not require stack gas scrubbing or limestone addition for sulfur retention; the quantity of oil produced is such that only light hydrotreating is required; the substitute natural gas is a direct product of hydrocarbonization and methanation is not required; and subbituminous coals are noncaking and do not agglomerate in the reactor.

In subsequent tests this technology is being applied to the bituminous coals of the East and Midwest. Chemical pretreatment has been found to render these coals noncaking and to facilitate the production of clean-burning char from high-sulfur coals.

**PLANS FOR THE COMING YEAR** — Exploratory studies with the existing bench-scale hydrocarbonization system will be conducted with the aim of identifying techniques for improvement of the hydrocarbonization process. Methods will be investigated for increasing the yield of oil by use of chemical pretreatment of the feed coal and by reduction of the vapor product residence time in the reactor. Improved technology for preventing agglomeration during hydrocarbonization of

bituminous coals will be tested, including the use of chemical pretreatment and internal char recirculation. Finally, low-pressure hydrocarbonization processes will be explored for production of low-sulfur char (a boiler fuel) in addition to oil and gas.

## GASIFICATION OF RESIDUAL MATERIALS FROM COAL LIQUEFACTION

TEXACO INC.  
DOE - \$929,840  
6/30/76 - 7/30/79

**OBJECTIVES** — The thermal efficiency of a coal liquefaction plant using a process that requires hydrogen or synthesis gas is dependent upon the efficient production of that gas from the non-liquefied fraction of the coal. This material, together with inorganic ash and some fraction of the converted coal, can be recovered after liquefaction as a pumpable residue. It has been demonstrated that high-ash coal-derived residues can be gasified directly using the Texaco Coal Gasification Process. In this work, the necessary design parameters will be determined for several residual materials resulting from DOE-sponsored liquefaction projects. All samples supplied by DOE will be evaluated initially in the laboratory to determine their suitability as feedstocks to the Texaco Coal Gasification Process. Large-scale pilot plant gasification runs will then be completed on promising materials to demonstrate operability, determine optimum operating conditions, and identify unexpected operating problems. The contract provides for a maximum of 15 laboratory evaluations and for a maximum of 16 weeks of large-scale pilot plant runs.

**RECENT WORK AND ACCOMPLISHMENTS** — Seven laboratory evaluations and one preliminary pilot plant evaluation have been completed to date. The materials tested are listed below.

| Material Tested                                     | Original Charge Coal | No. of Samples | Test Level* |
|---|----------------------|----------------|-------------|
| H-Coal Syncrude Vacuum Tower Bottoms                | Ill. No. 6           | 1              | I           |
| H-Coal Settler Stripped Underflow                   | Ill. No. 6           | 1              | I           |
| Synthoil Centrifuge Underflow                       | Kentucky             | 1              | I           |
| USS Clean-Coke Hydrogenation Vapor Stripper Bottoms | Ill. No. 6           | 1              | I           |
| H-Coal Syncrude Vacuum Tower Bottoms                | Ill. No. 6           | 1              | II          |
| SRC-II Vacuum Flash Drum Bottoms                    | Kentucky             | 1              | I           |
| EDS Vacuum Tower Bottoms                            | Ill. No. 6           | 1              | I           |

\*Test Level I - Laboratory Evaluation of ~5-gal sample.

Test Level II - Preliminary Pilot Plant Evaluation of ~20-bbl sample.

All of the residues, except the H-Coal Settler Stripped Underflow, were considered to be suitable candidate feedstocks for the Texaco Coal Gasification Process. Large-scale pilot plant tests with residue from the H-Coal, SRC and EDS processes are feasible and can be carried out at DOE's option whenever enough material is available. As presently equipped, large-scale pilot plant tests with residues from the SRC II and Exxon Donor Solvent processes may require the use of a suitable cutter stock to reduce the viscosity of each residue at the limited operating temperatures achievable in the pilot plant. About 4½ tons of H-Coal Syncrude Vacuum Tower Bottoms from the liquefaction of Illinois No. 6 coal were gasified in a single 9-hr pilot plant run at 350 psig. A 97 percent conversion of the carbon in the feed to syngas was achieved yielding 32.6 scf of dry syngas per pound

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of residue gasified. No significant operating problems were encountered. The dry syngas produced contained over 93 volume percent carbon monoxide and hydrogen.

**PLANS FOR THE COMING YEAR** – Provisions have been made for the three types of tests:

|   | Max.<br>Sample<br>Size | Min.<br>No. of<br>Samples | Max.<br>No. of<br>Samples |
|---|------------------------|---------------------------|---------------------------|
| Type 1 Laboratory Evaluation              | 20 lb                  | 4                         | 15                        |
| Type 2 Preliminary Pilot Plant Evaluation | 20 bbl                 | 0                         | 4                         |
| Type 3 Extended Pilot Plant Evaluation    | 200 bbl                | 0                         | 4                         |

All materials received will undergo a Type 1 evaluation. Each succeeding type of evaluation will be performed only on the most promising materials from the preceding evaluation at the request of DOE.

#### COAL MINE-COAL CONVERSION PLANT INTERFACE

BECHTEL CORPORATION

DOE - \$601,593

6/76 - 11/77

**OBJECTIVES** – Major project objectives were investigation of operations which constitute the coal mine-coal conversion plant interface, determination of any technology limitation, development of conceptual designs for selected complete interfaces, and estimation of capital and operating costs for selected interfaces. An additional objective was evaluation of probable impact of large coal conversion plants on regional transportation networks. New concepts that could improve interface economics or be required to meet future environmental regulations were also identified. The project final report provides DOE and its contractors with the information required to develop conceptual designs and cost estimates for numerous different interfaces.

**RECENT WORK AND ACCOMPLISHMENTS** – The interface project was confined to consideration of mine-conversion plant interfaces that could be required for the Appalachia coal region, but the information resulting from the project is applicable to other coal producing regions. Two general coal conversion plant site alternatives were considered: an integrated coal mining/coal conversion site which obtains its entire coal requirement from an adjacent, contiguous integrated mining complex, and a regional coal mining/remote coal conversion site which obtains its coal from many regional mines located appreciable distances from it. Coal would be transported to the remote plant by rail or barge or a combination of these methods. To aid in project results presentation, a reference interface design was defined. This complete interface includes design modules which cover receiving and breaking of coal produced by an underground mining complex; coarse coal live and dead storage; coal and limestone conveying (limestone is used for power plant flue gas desulfurization); simultaneous coal pulverizing/drying; steam and coal pulverizing/drying gas generation by fluidized-bed combustion; power generation and process steam distribution; and, solid wastes collection, transport, and disposal. Alternative interface design modules allow evaluation of interfaces for conversion plants which obtain all or part of their coal from regional mines by barge

and rail transport, require totally enclosed coal storage, use coal cleaning, and which use alternative coal pulverizing systems. Estimated total capital cost of the complete reference interface (1977 \$) is \$759.1 million excluding cost of the integrated coal mining complex. Estimated engineering-construction duration for this interface design is 6 years. Reference interface estimated mean annual operating cost is \$87.9 million including depreciation. Estimated order-of-magnitude capital cost for the underground mining complex postulated for the reference interface is \$426.1 million and its estimated annual operating cost is \$126.7 million.

**PLANS FOR THE COMING YEAR** – This project was completed in 1977.

## ENGINEERING STUDIES AND EVALUATIONS

OAK RIDGE NATIONAL LABORATORY  
DOE - \$741,000  
1975 - Continuing

**OBJECTIVES** – Technical and economic evaluations of coal conversion processes will be made. These evaluations are designed to provide estimates of the costs of producing synthetic fuels from coal on a commercial scale, to assess the state of development and technical feasibility of the process under consideration, and to provide recommendations for additional process or equipment research and development where needed.

**RECENT WORK AND ACCOMPLISHMENTS** – Final reports on evaluations of the Synthoil and hydrocarbonization processes were issued. Each evaluation was based on a complete process design for a grass roots facility. The designs included flow diagrams, mass and energy balances, equipment sizing, and cost estimates. The size of the plant chosen as the basis for the evaluation in each case was such that the total heating value of products was equivalent to the production of 100,000 bbl of fuel oil per day. The Synthoil process, currently under development at Pittsburgh Energy Research Center (PERC), is a fixed-bed, catalytic hydrogenation process for the production of desulfurized fuel oil from coal. The design basis for the 100,000-bbl/d commercial facility used in the evaluation was derived from experimental data obtained at PERC.

The estimated overall capital investment for the facility was \$1897 million, consisting of \$1688 million depreciable and \$229 million nondepreciable capital. All costs were estimated on first-quarter 1976 dollars. During the discounted cash flow (DCF) method, the price of product oil was calculated as a function of coal cost and annual aftertax rate of return on equity capital. Several cases were calculated to provide sensitivity information. As an example of the results, it was found that assuming 100 percent equity capital, 12 percent aftertax return on equity, and a coal cost of \$20/ton, the price of product oil was about \$30/bbl or \$4.67/million Btu.

A hydrocarbonization system based upon the U.S. Steel "clean coke" process was selected for detailed evaluation after extensive reviews of the literature on coal carbonization and hydrocarbonization. The final report includes the conceptual design and cost estimate of a commercial facility for producing clean char, oil, and pipeline gas fuels having a heating value comparable with 100,000 bbl/d. The conceptual design includes a complete facility. The total capital investment, including initial working capital, initial raw materials, catalysts and chemicals, and land, was estimated at \$1019 million. A DCF analysis, assuming 100 percent equity financing, 12 percent return on equity, and coal priced at \$20/ton, resulted in an overall product fuel price of

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\$3.15/MMBtu. Results of the study emphasize the need for testing the process in a continuous pilot plant using a wide variety of highly caking bituminous coals as feed material. A program suggested for the pilot plant would encompass development of improved methods for the prevention of agglomeration of highly caking coals during hydrocarbonization, optimization of the yields of coal liquids, investigation of a single-stage, high-temperature hydrocarbonizer optimized for char production, and optimization of beneficiation ratios employed during coal preparation.

**PLANS FOR THE COMING YEAR** — Additional studies will be initiated as requested by DOE.

## POTENTIAL COMMERCIAL COAL CONVERSION PROCESSES

F. KUNREUTHER ASSOCIATES, INC.

DOE - \$85,180

6/75 - 10/77

**OBJECTIVES** — This project is designed to (1) provide simple low-cost engineering analyses and evaluations of process alternates for selected coal conversion plant processes, or proposed plant designs applying all aspects of fuels technology; (2) investigate independently proposed concepts and plant processes, and evaluate technical cost and schedule tradeoffs; (3) perform analyses to identify weaknesses, or validate total plant concepts; and (4) identify technical areas, wherein plant process can be improved by substitution of process alternatives.

**RECENT WORK AND ACCOMPLISHMENTS** — Work performed under Task I, technical feasibility and process integrity of the Consol synthetic fuel process, concluded that the coal extraction step could be simplified and made more reliable by eliminating staging and mechanical mixing. Solvent and liquid products should be recovered by vacuum flashing and fixed-bed hydrogenation rather than hydroclones and ebullating bed hydrogenation. Extraction temperature should be increased to make this feasible. Fluid coking and possibly Flexicoking should be investigated along with low-temperature carbonization of heavy residue and undissolved coal. Hydrogen manufacture should not include boosting hot char from low to high pressure. Under Task 2, gasoline from coal extract, a flow scheme was developed for converting coal extract from the Consol synthetic fuel process to gasoline and lighter hydrocarbons. It was determined that 3.4 bbl of 100 R-O gasoline could be produced per ton of MAF coal to extraction with another 0.3 tons MAF coal required for fuel. Three-stage hydrogenation followed by hydrocracking, and mild catalytic reforming was found suitable for achieving the above yields, if the extract feed is a distillate free of solids and dissolved ash components. Hydrogen should be produced from gas generated in the process plus partial oxidation of nonvolatile extract slurry. Investment cost for the above extract conversion process was estimated at \$7000 per daily barrel of gasoline.

Under Task 3A, production of gasoline from coal extract by catalytic cracking, it was found that severe hydrogenation of coal extract followed by catalytic cracking appears to be technically and economically a more attractive route to gasoline from coal than hydrocracking. Further study is suggested to determine the catalytic cracking advantage quantitatively. Under Task 3B, comments on process flow sheets for advanced coal liquification plant developed by Fluor Corporation, the flow sheets of eight units were critically reviewed from a technical and operational point of view. Under Task 3C, hydroprocessing of coal and shale derived liquids: causes of catalyst activity decline and methods for improving catalyst life, it was concluded that strict exclusion of air from products destined for catalytic hydroprocessing is required. In addition, catalytic pretreatment at high



hydrogen pressure and low reactor temperature is required to eliminate highly unstable feed components.

**PLANS FOR THE COMING YEAR** – Project tasks were completed in 1977.

### **RESIDUE CARBONIZATION**

**OAK RIDGE NATIONAL LABORATORY**

DOE - \$66,000

10/1/74 - Continuing

**OBJECTIVES** – Existing carbonization facilities will be utilized for carbonization tests of coal liquefaction residues as requested by DOE. One series of tests is aimed at estimating the recovery of volatiles from the atmospheric pressure carbonization of H-Coal vacuum still bottoms. A second series of tests is aimed at assessing the influence of pressure, up to 4000 psi, on solvent losses during carbonization of CSF hydroclone or settler underflows. A third series of tests is aimed at estimating recovery of volatiles from carbonization of H-Coal vacuum still bottoms mixed with coal under rapid pyrolysis conditions in methane at pressures to 400 psi.

**RECENT WORK AND ACCOMPLISHMENTS** – Residue carbonization tests were completed in response to three requests from DOE. When the vacuum still bottoms from H-Coal were carbonized in a fluidized bed at atmospheric pressure and temperatures to 1200°F, only negligible quantities of volatile liquids were recovered. Serious and unresolved problems were encountered in carbonizing this feed material in a fluidized bed as a result of agglomeration in the bed and plugging of downstream equipment. When CSF hydroclone or settler underflow was carbonized at elevated pressure, solvent losses were essentially unaffected. Minor solvent losses at 900° to 1200°F were unchanged by pressures to 400 psig. Results from the third series of tests, involving rapid pyrolysis under methane pressure of coal plus H-Coal vacuum still bottoms, are still incomplete.

**PLANS FOR THE COMING YEAR** – Results from the tests described will be presented in a final report. No further experiments are planned, but the facilities will be maintained for future tests that may be required.

### **COAL LIQUEFACTION SUPPORT STUDIES**

**ARGONNE NATIONAL LABORATORY**

DOE - \$972,000

7/76 - 9/77

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**RECENT WORK AND ACCOMPLISHMENTS** — A calorimetric system, to be used to measure heats of reaction between hydrogen and coal slurries under coal liquefaction process conditions (350° to 475°C, 1000 to 3000 psia hydrogen pressure), has been built. A test unit has been fabricated to measure heat transfer coefficients between coal slurry, preheat and reactor effluent fluids, and metal surfaces. Shakedown tests have been made with hydrogen and hydrogen-creosote mixtures, and the equipment has functioned satisfactorily. A continuous high-pressure coal-liquefaction unit (catalyst test unit) has been constructed to operate at coal-liquefaction process conditions—450°C and 1000 to 4000 psi hydrogen pressure.

**PLANS FOR THE COMING YEAR** — Work on the project was suspended September 30, 1977, pending reevaluation of coal liquefaction priorities. Equipment will be kept available for making needed measurements on heats of reaction and coefficients of heat transfer.

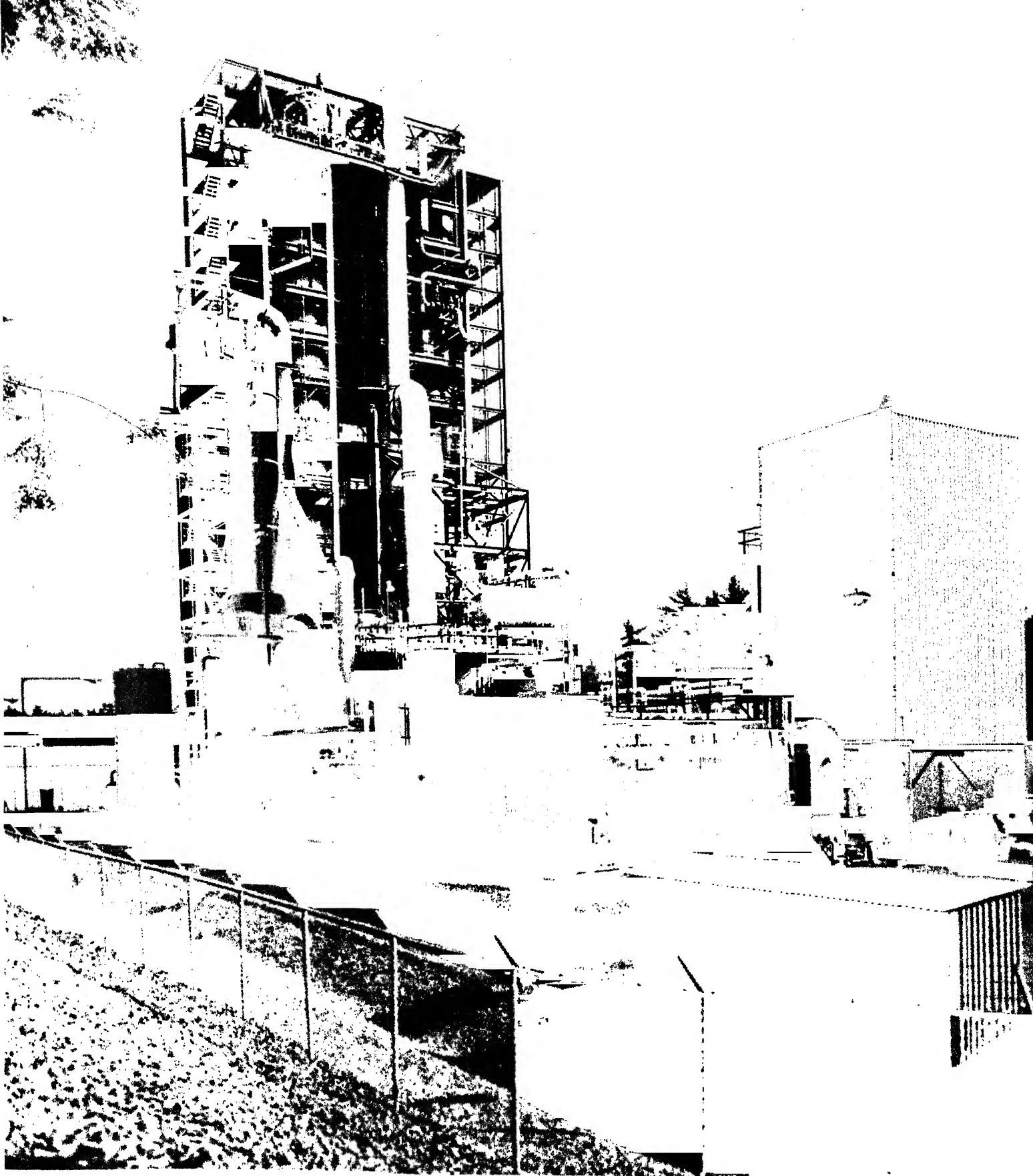
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*Combustion Engineering 5-T/Hr Coal Gasification Process Development Unit*

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# ***GASIFICATION***

Both high- and low-Btu gasification processes are being developed by the Department of Energy. High-Btu gas can be distributed economically to consumers in the same pipeline systems now used to carry natural gas. Low-Btu gas, the cheapest of the gaseous fuels produced from coal, can be used economically only on site, either for electric power generation or by industrial plants.

## **HIGH-BTU GASIFICATION**

DOE and the American Gas Association are jointly funding the development of a number of the processes for converting coal to high-Btu gas. These processes are at the pilot plant stage of development. Responsibility for designing, constructing, and operating these pilot plants is assigned to Conoco Coal Development Company for the carbon dioxide acceptor pilot plant at Rapid City, South Dakota; Bituminous Coal Research, Inc., and Phillips Petroleum Company for the Bigas pilot plant in Homer City, Pennsylvania; Institute of Gas Technology for the Hygas pilot plant and steam-iron system for the production of hydrogen in Chicago, Illinois; and the Lummus Company for the Synthane pilot plant in Allegheny County, Pennsylvania. Battelle Memorial Institute is responsible for designing, constructing, and operating a process development unit in West Jefferson, Ohio, for demonstrating the agglomerating burner process. These companies are also responsible for continued laboratory research to develop data for verifying the feasibility of the specific process and for supporting the operation of the plant.

A liquid phase methanation process is being developed by Chem Systems, Inc., while C.F. Braun and Company is providing technical evaluations of these high-Btu gasification processes and developing conceptual designs of commercial plants for producing pipeline-quality gas as a basis for evaluating the technical and economic feasibility of each.

The development work on the CO<sub>2</sub> acceptor coal gasification process was completed during 1977. The experimental data are being correlated and evaluated with final reports in preparation. The facility has been closed down and placed on a standby basis while other projects are being evaluated as they might be tested in the facility. Test operation of the pilot plant for the Bigas process for the generation of pipeline gas was started. Subbituminous coal from Montana's Rosebud Mine was utilized for startup of the pilot plant. Laboratory research continued to focus on experiments to develop further the Bigas process and the equipment required with emphasis on fluidized-bed catalytic methanation.

Technical evaluations of the progress being made by various DOE contractors and specific aspects of high-Btu gasification are continuing as is development of conceptual designs of commercial gasification plants. Operations were observed at the CO<sub>2</sub> acceptor, Bigas, Hygas, and Synthane pilot plants to obtain data for the conceptual design of commercial-size plants for each candidate process. The Hygas steam-iron process using western coal was revised. Preparation of process flow diagrams and determination of equipment requirements for the CO<sub>2</sub> acceptor process using lignite, and the Bigas, Hygas steam-oxygen, and Synthane processes using eastern coal were continued.

## LOW-BTU GASIFICATION

Low-Btu gas, with a heating value of 120 to 500 Btu/ft<sup>3</sup>, is an economical fuel for power generation in combined gas-steam turbine power cycles and as an industrial gaseous fuel. On an equivalent Btu basis, conversion of coal to low-Btu gas is less complex than to high-Btu gas and the capital costs are lower. Because different low-Btu gasification processes are optimum for converting different types of coal and because of the need to provide commercially acceptable processes at the earliest possible date, DOE is sponsoring the concurrent development of several basic types of gasifiers (fixed-bed, fluidized-bed, and entrained-flow). At the same time, hot-gas cleanup systems are being studied for use with these low-Btu gasifier systems.

Responsibility for designing, constructing, and operating facilities for testing low-Btu gasification processes has been assigned to Atomics International Division for a molten salt gasification process; Westinghouse Electric Corporation for an advanced coal gasification system for electric power generation; Combustion Engineering, Inc., for low-Btu gasification of coal for generating electricity and providing industrial fuel; and Bituminous Coal Research, Inc., for a low-Btu fuel gas process. In each case, laboratory research is also being conducted to resolve process problems and improve overall process designs.

Air Products and Chemicals, Inc., is conducting a support program involving desulfurization of low-Btu producer gas in a fixed bed of iron oxide on fly-ash; Gilbert Associates, Inc., is providing technical and engineering services; and a technical data book is being developed by the Institute of Gas Technology to provide a single, comprehensive source of data on coal conversion processes.

Since low-Btu gas must be generated at its point of use, actual integration of production and end-use application is a critical element in establishing its utilization. Three major programs are in progress to develop industrial and utility applications. *Gasifiers in Industry* involves the design, construction, and operation of small, state-of-the-art gasifiers in cooperation with industry. This project will provide data from industrial operation as an input to the development of new gasification processes. As a result of a Program Opportunity Notice (PON), six proposals were selected for negotiation. Contracts have been negotiated. Accurex-Aerotherm (in conjunction with Glen Gary Brick Company) is installing a fixed-bed gasifier for the use of anthracite coal to fire a brick kiln with low-Btu gas. The University of Minnesota will utilize low-rank coal to produce low-Btu gas for supplying fuel to the University's Duluth campus steam plant. Additional end uses will include a dairy plant, housing community, refractory plant, and industrial park. In all cases, 50 percent of the cost of these projects is being funded by the industrial proponent. *The Coal to Hydrogen Facility* will demonstrate the production of hydrogen from coal. Proposals for the facility are being evaluated. These proposals in response to a PON will be presented to the selection official in early April. *The Coal-Gasification Combined-Cycle Test Facility* is planned to provide a meaningful evaluation of the use of low-Btu gas for the generation of power. Negotiations were completed for the construction and operation of this facility with a major electric utility and the Electric Power Research Institute.

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# ***HIGH-BTU GASIFICATION***

## **PIPELINE GAS DEMO PLANT PROGRAM**

**ILLINOIS COAL GASIFICATION GROUP**  
DOE - \$157,954,000; Illinois Coal - \$135,262,000  
6/7/77 - 7/7/85

**OBJECTIVES** – The commercial feasibility and economic attractiveness of the COGAS process will be demonstrated for conversion of high sulfur coal to clean pipeline gas. The COGAS process converts coal to both gas and liquid products by taking advantage of the high efficiency of pyrolysis of the coal and gasifying the resultant char to produce synthesis gas in a low-pressure system without the requirement of bulk oxygen. Specific objectives include developing a conceptual design for a future commercial sized plant (250 million scf/d of pipeline quality gas), developing a process design for a demonstration plant, engineering a definitive design of a demonstration plant capable of converting approximately 2,000 dry t/d of Illinois No. 6 coal (or either one or both of two alternate coals) to gas and oil, constructing the demonstration plant on a southern Illinois site, and finally operation of the demonstration plant to prove the commercial feasibility and economics of coal gasification using the COGAS process. The use of the COGAS process offers the potential for economical and efficient production of pipeline gas and oil from this Nation's abundant reserves of coal, thus supplementing the decreasing supply of natural gas and oil and decreasing the reliance on imported gas and oil.

**RECENT WORK AND ACCOMPLISHMENTS** – A tentative baseline design for the commercial plant process concept has been completed. Trade-off studies of various subprocesses (e.g., sulfur recovery, gas purification, etc.) are being conducted to determine which ones will best complement the COGAS process to provide the best demonstration plant process design and commercial plant conceptual design. Physical work being accomplished with regard to site selection and evaluation includes taking soil borings, making topographical and land surveys, and doing environmental sampling. Additionally, pilot plant tests, cold model tests, and laboratory experiments have continued to provide detailed design information and parameters.

**PLANS FOR THE COMING YEAR** – Work will include completion of process analyses for all subprocesses, a commercial plant conceptual design, site selection and evaluation, demonstration plant process design and recommendations, and pilot plant studies. Work will be initiated on the definitive design of the demonstration plant and construction planning. Environmental analysis work will be completed, and work will start on the environmental impact statement.

## **PIPELINE GAS DEMO PLANT PROGRAM**

**CONTINENTAL OIL COMPANY**  
DOE - \$198,344,000; Continental - \$173,244,000  
6/1/77 - 9/1/85

**OBJECTIVES** – This project will demonstrate commercial feasibility and economic attractiveness of a chemical process for the conversion of high-sulfur caking coal to clean pipeline gas. This program includes the following specific objectives: (1) to engineer, design, construct, operate and



manage an integrated synthetic pipeline gas demonstration plant; (2) to confirm design criteria, demonstrate technology, and obtain experience which will yield those characteristics required to establish and/or define plant investment and operating costs for future clean pipeline gas commercial production plants; (3) to project plant investment, operating, and end product costs for full-scale commercial production plants; (4) to provide technology, performance, reliability, and maintenance characteristics which will permit the design of a full-scale clean pipeline gas commercial production facility; (5) to investigate the general applicability and limits of the selected process and plant design for a variety of coal types; (6) to demonstrate scaling parameters (with respect to the commercial production of the primary product) and designing of major process components; (7) to provide finished marketable clean pipeline gas in quantities sufficient for end use evaluations; (8) to determine the general characteristics, composition, and quality of the end products; (9) to assess and/or evaluate characteristics and suitability of the product in actual representative end use applications; (10) to establish guidelines for by-product use and/or disposal; (11) to obtain data which will permit design of an environmentally acceptable commercial plant; and (12) to establish the design compatibility with Occupational Safety and Health (OSHA) requirements.

The Conoco/DOE project will demonstrate the technology of the British Gas/Lurgi slagging gasifier in the conversion of a moderately caking coal. This gasifier, operating at temperatures high enough to melt the coal ash, is theoretically more efficient than the existing Lurgi dry bottom gasifier. The development of coal gasification technology will permit the U.S. to utilize its abundant coal reserves to replace the dwindling supplies of natural gas and oil. While creating a more convenient form of fuel for the consumer, gasification also facilitates the removal of sulfur and other potential polluting elements from the coal.

**RECENT WORK AND ACCOMPLISHMENTS** – This project is divided into three phases: development and engineering, demonstration plant construction, and demonstration plant operations. Phase I of this project was started on July 1, 1977. Design work is currently underway for a hypothetical commercial plant, using the slagging gasifier and Illinois No. 6 coal. The process design work for the demonstration plant and the gasifier has been started using Ohio No. 9 coal as the feed. A location for the demonstration plant has been recommended in Noble County, Ohio, and site characterization work has begun. The environmental studies have been started, and the first season's monitoring program is complete. Concurrent with the design effort, a technical support program is being performed at BGC's Westfield Development Center in Scotland. Ohio No. 9 coal is being gasified in a semiworks-scale slagging gasifier to verify assumptions relating to the operating conditions and design data for the demonstration plant. Pittsburgh Seam 8 coal, a highly caking coal, will also be tested at the Westfield Works.

**PLANS FOR THE COMING YEAR** – The commercial plant design, the demonstration plant process design, the technical support program at Westfield, and the site evaluation will be completed by July 1978 for review by DOE. The design, planning, and evaluation effort will be completed by the end of FY 1979. Construction of the demonstration plant will start soon after completion of Phase I.

**HYGAS DEMO PLANT PROGRAM**

PROCON INC.  
DOE - \$7,540,196  
7/29/77 - 3/29/79

**OBJECTIVES** – The HYGAS Demonstration Plant Project is a part of the overall high-Btu gasification program. The total program objectives are to verify the technical and economic feasibility of converting coal to substitute natural gas (SNG) of pipeline quality, and to provide an environmentally acceptable plant that can utilize all types of coal. Eventually, this program will supplement natural gas supplies and reserves by gasifying coal on a commercial scale. The current HYGAS conceptual design project has two distinctive parts, the first one being the conceptual design and economic evaluation of a commercial-size plant for production of substitute natural gas (SNG) utilizing the Institute of Gas Technology (IGT) HYGAS Coal Gasification Process; the second consists of the development of a partly detailed process design for a demonstration plant, with components large enough to provide solutions to problems that might be met in a commercial-size plant including utility and off-site facilities. This detailed process design will be based on a generic site. The basic objective of the commercial design is the development of the cost of the SNG produced in a  $250 \times 10^6$  scf/d plant. The economic analysis will require the development of the process design, equipment sizing, and other engineering work in sufficient detail to produce a budget-type cost estimate. The demonstration plant, with a capacity yet to be determined, will have the capability of being expanded to full commercial size. As the A/E, Procon will develop a process design package suitable as a basis for completion of detailed engineering, procurement, and construction by others. A cost estimate and a complete economic analysis will also be prepared for the demonstration plant.

**RECENT WORK AND ACCOMPLISHMENTS** – During the first 2 months of the project, a base case commercial plant design was developed. Alternatives for various units within the plant were identified for comparative studies. The base case design began with preparation of an overall process description. A recommendation of design coal feedstocks was prepared. A range of representative feedstocks will be considered including western lignite and Illinois Basin bituminous coal. Design criteria were developed. An overall material balance and block flow diagrams were prepared.

**PLANS FOR THE COMING YEAR** – The commercial plant conceptual design and cost estimate will be completed. The cost of gas from the plant will be determined. An economic analysis report of the HYGAS concept will be prepared. The demonstration plant design will proceed through the basic process engineering phase, and mechanical component and system design will have begun.

**CO<sub>2</sub> ACCEPTOR COAL GASIFICATION PROCESS**

CONOCO COAL DEVELOPMENT COMPANY  
DOE - \$47,562,370; American Gas Association - \$10,294,000  
6/64 - 12/77

**OBJECTIVES** – This process for coal gasification is being developed under the joint sponsorship of DOE and the American Gas Association. The principal objectives of this effort are to demonstrate the operability of all process features and thus establish that its comparative advantages are real and can be incorporated into the design of a commercial coal gasification facility. These advantages

include the ability to produce synthesis gas without the need of an oxygen plant, production of a synthesis gas that has a high  $H_2/CO$  ratio and is therefore suitable for methanation without the need of a shift-conversion unit, high carbon utilization, and minimization of cleanup facilities because of the lack of oils and tars and the low concentrations of  $CO_2$  and  $H_2S$ . The process is especially well suited for gasification of the enormous reserves of Western lignites and subbituminous coals.

**RECENT WORK AND ACCOMPLISHMENTS** – The pilot plant experimental program was concluded September 30, 1977. During its operational period, all the original goals of the program were accomplished. These operational achievements are summarized as follows. The  $CO_2$  Acceptor pilot plant has operated over the period from April 1972 through August 1977. Over 79 separate runs were completed. The plant was at normal operating conditions of pressure and temperatures for 6400 hours, and 6500 tons of dry coal were gasified. Process conditions are defined as: the acceptor is circulating continuously, both the gasifier and regenerator are at programmed temperatures, combustion of char is supplying all the regenerator heat duty, and the circulating acceptor is supplying all or part of the gasifier heat duty. During this period several coals were gasified successfully. These include three North Dakota lignites—Velva, Glenharold, and Husky; one Texas lignite; and three subbituminous coals—Sarpy Creek and Rosebud (Montana), and Wyodak (Wyoming). Detailed operating data on the process were obtained with the Velva, Glenharold, Texas, and Wyodak coals. Plant operations at steady conditions produced 12 heat and material balances. The length of runs covering these operations varied from 130 to 290 hours at process conditions of pressure and temperatures. During the period from January through August 1977, nine runs were made and the pilot plant was on stream for 40 percent of the time. The acceptor supplied the entire gasifier heat during 70 percent of that time.

Both the regenerator and gasifier were relined with refractory materials early in the pilot plant program to correct a design deficiency. Since then the gasifier refractory has been in use for 64 runs. In each run, a complete temperature-pressure cycle occurred, starting and ending with an open, cold reactor with operation at full process pressure and temperature in between. In the same period the regenerator, which operates at a much higher temperature than does the gasifier, has had three linings. The first lining had 23 temperature-pressure cycles as defined above, and the second lining had 30 cycles. The third and current lining was installed to reduce the working diameter. It has undergone nine cycles and is in excellent condition. There has been no appreciable interaction of either refractory system with the acceptor or with coal ash. The pilot plant experience clearly shows that a large plant, with infrequent cycling, will have no fundamental problems with refractories.

An intensive sampling and analytical program has been completed that will lead to comprehensive definition of all process streams and effluent streams in the pilot plant. Although final data workup is not yet complete, it appears that the envisioned environmental controls for the commer-

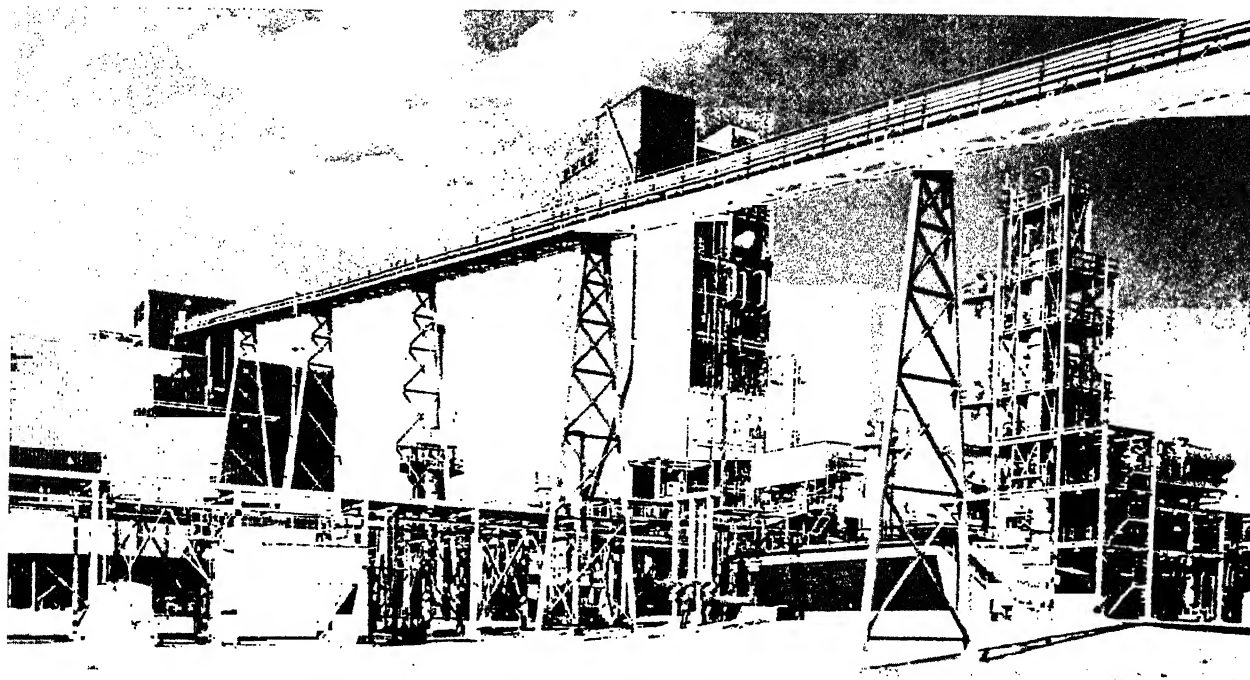
## GAS GENERATOR RESEARCH AND DEVELOPMENT—BIGAS PROCESS

BITUMINOUS COAL RESEARCH, INC.  
DOE - \$55,799,682; American Gas Association - \$18,761,940  
8/19/71 - 12/31/78

**OBJECTIVES** — As a result of extensive laboratory research and testing at a process and equipment development unit (PEDU) scale involving capacities of 100 lb/hr of coal, the BIGAS process demonstrated a potential for production of high-grade synthesis gas from a wide range of coals. In addition, laboratory investigations indicated the advantages of fluidized-bed methanation over fixed-bed systems for upgrading the calorific value of a coal-derived synthesis gas. These R&D efforts at PEDU scale provided the basis for design and construction of a complete pilot plant facility with a throughput capacity of 5 t/hr of coal. The primary objective of the current program is to develop the BIGAS coal gasification process further by demonstrating operability of a complete pilot plant facility. Emphasis is on the operation of coal preparation and feeding systems, an entrained slagging gasifier, and a fluidized-bed methanation system. The ultimate objective is to obtain data sufficient for design of a commercial-size plant for production of SNG from coal by the BIGAS process.

**RECENT WORK AND ACCOMPLISHMENTS** — Construction of the pilot plant facility at Homer City, Pennsylvania, was completed in mid-1976 by Stearns-Roger Inc. Stearns-Roger is also responsible for plant operations and maintenance. Phillips Petroleum Company, as the pilot plant management subcontractor, has overall responsibility for planning and conducting the pilot plant research program. After construction, the plant was completely staffed and a comprehensive operator training program was pursued. Systems checkout activities, begun in March 1976, were completed by December. Step-by-step shakedown was required in several individual process areas because many systems had never been tested under the high-pressure and high-temperature conditions of the BIGAS process. Natural gas curtailment during the first 2 months of 1977 delayed full operation of the gasifier until mid-March. During 1977, efforts were directed toward operation of the gasifier and the slurry preparation and coal flash-drying systems that precede it. Numerous mechanical failures of plant components and the inability to coordinate the flow of materials (coal, char, slag) were encountered during the period. Whole solids flow measurement and the ability to determine temperatures accurately in the slagging stage of the gasifier remain problems to be solved, they are currently receiving major attention. A qualified environmental services subcontractor has been engaged in accumulating data on plant effluents and ambient air quality. These data will be used to assess the environmental impact of the BIGAS process. Modeling studies with JAYCOR and kinetic studies at MIT were initiated to develop data for use in establishing a more thorough understanding of gasifier performance.

Activities in direct support of the pilot plant continued at the BCR facilities. Through use of a high-temperature slag viscometer, viscosity data were obtained on Montana Rosebud coal ash and on the effect of flux additions to that ash. These data serve to guide pilot plant personnel in attempts to alter operating conditions to assure proper tapping of the slagging stage of the gasifier. Catalysts for use in the fluidized-bed methanation system continued to be evaluated. After a series of bench-scale tests were performed, certain catalysts were elected for more intensive testing in the methanation PEDU. The PEDU permits intermediate-scale testing and provides data that can be used to establish optimum conditions for operation of the larger, pilot-scale unit. The catalyst that was selected as the initial charge for the methanation system in the BIGAS pilot plant was subjected to additional testing in anticipation of the impending activation of the pilot unit.



*Bi-Gas Coal Gasification Pilot Plant-Homer City, Pennsylvania*

**PLANS FOR THE COMING YEAR** – Research activities at the pilot plant will emphasize integrated operation of all unit processes, and gasification of several different coal types. Complete investigation of all process variables will permit establishment of optimum operating conditions for the BIGAS process. In support of the pilot plant programs, research will continue on evaluation and optimization of operations with fluidized-bed methanation catalysts. Additional testing will be conducted to determine the feasibility of combining the shift and methanation reactions in a single process step.

#### **AGGLOMERATING BURNER GASIFICATION PROCESS**

**BATTELLE, COLUMBUS LABORATORIES**  
DOE - \$14,810,490; American Gas Association - \$569,395  
1/2/73 - 1/31/78

**OBJECTIVES** – The current program plans to demonstrate basic operability of the 25-ton/d Process Development Unit (PDU) and then to develop the process to be economically attractive and commercially available. The immediate objective is to operate the PDU in an integrated fashion for an extended time. Equipment modifications and improvements have been and continue to be made to advance the PDU toward fulfilling these objectives. Materials and equipment testing work is continuing as a fundamental part of developing this technology. A turbine blade erosion rig is to be tested in operation to further the design of a power recovery gas turbine.

**RECENT WORK AND ACCOMPLISHMENTS** – PDU operation has demonstrated solids circulation rates and control over the range of 10-30 tons/hour. Coal combustion studies have yielded a control freeboard burning with fine coal (-100 mesh). Agglomeration of subbituminous (Rosebud) coal was demonstrated with 90 percent capture of the ash. Coal preparation and handling systems have been improved to provide satisfactory coal feed to both the combustor and the gasifier. The process

operating pressure has been increased from 60 to 100 psig by debottlenecking equipment. The off-gas scrubber systems have been modified to permit control of the process during upset conditions. All utilities and support systems have been worked to provide reliable service with a satisfactory service factor.

**PLANS FOR THE COMING YEAR** — This project is being reevaluated.

### SYNTHANE PROTOTYPE PILOT PLANT

THE LUMMUS COMPANY

DOE - \$31,990,000

8/1/74 - 9/30/78

**OBJECTIVES** — The Synthane Pilot Plant Program is designed to obtain definitive process information, sufficient for commercial design purposes, for the conversion of coal to pipeline-quality gas. This objective is to be accomplished through the execution of experimental and developmental operation programs in the government-owned Synthane pilot plant, located in South Park Township, Allegheny County, Pennsylvania. Specific objectives are to operate the pilot plant and to verify the economic and technical feasibility of the Synthane process over a wide variety of coal types. In view of the limited supply of natural gas within the United States and the projected future requirements, the Synthane process is being evaluated and demonstrated as a means of supplying supplemental fuel. The process offers an excellent potential for the commercial production of pipeline-quality synthetic natural gas and medium-Btu gas because of its basic simplicity, concept, and overall projected efficiency.

**RECENT WORK AND ACCOMPLISHMENTS** — The Synthane pilot plant began gasification in July 1976, with a western subbituminous noncaking Rosebud coal. Since that date, the plant has processed over 2000 tons of Rosebud coal and produced gas for a total of 800 hours. Much of the first year was devoted to achieving operating reliability through modification of equipment and operating procedures. During August 1977, Rosebud coal was successfully gasified for a period of 190 hours, 98 hours of which were continuous. For extended periods, design carbon conversions of better than 75 percent to 80 percent were achieved and sufficient data were achieved to provide a basis for a commercial design for this type of coal. Based upon the actual plant data from this run, a detailed economic assessment of the Synthane process for gasification of western subbituminous Rosebud coal was made by The Lummus Company. The study projects gas production from a commercial facility at \$3.40/MMBtu. The success of the August 1977 run effectively terminated studies with Rosebud coal and achieved the test directive requirements as detailed by DOE. The plant is presently operating using a mildly caking subbituminous Illinois No. 6 coal which requires pretreatment before gasification. The Synthane plant offers a continuous on-line pretreatment design which is currently being tested.

**PLANS FOR THE COMING YEAR** — Pilot plant studies will continue with the gasification of Illinois No. 6 coal. The program will be extended to include other eastern coals as specified by DOE. Thus far, only those facilities necessary for the testing of the gasifier have been operated. The commissioning of all remaining gas purification facilities will be achieved within the year. More emphasis will also be placed on the collection of equipment reliability data to be available for consideration in a commercial design.

## STEAM-IRON PROCESS FOR HYDROGEN PRODUCTION

INSTITUTE OF GAS TECHNOLOGY

DOE - \$22,909,000; American Gas Association - \$11,450,400\*

5/1/73 - 9/30/77

**OBJECTIVES** — The Steam-Iron Program is designed to develop an economically attractive method for producing hydrogen from coal. Almost all of the hydrogen presently produced in the United States is made by reforming of natural gas. Because of the ever-increasing shortage of natural gas, new methods of producing hydrogen from vast coal reserves are needed. Furthermore, the production of hydrogen is a key step in the conversion of coal to clean-burning sulfur-free synthetic gas and oil. The steam-iron process may be useful in many fossil fuel conversion processes now under development by DOE. It may also be useful in ammonia manufacture, petroleum refining, alcohol manufacture, and metallurgy. A definitive pilot plant test program will be conducted to verify the technical and economic feasibility of the steam-iron process for hydrogen production and to provide detailed data for the design of commercial plants. Specific objectives of this project have been to start-up and test a pilot plant designed to produce 1.1 million scf of hydrogen daily. The reactor system of this pilot plant comprises two principal vessels, a producer reactor and a steam-iron reactor. In the producer reactor, a reducing gas is made by reacting coal char with steam and air. In the steam-iron reactor, iron ore is continuously circulated and cyclically reduced by the producer gas and oxidized with steam to produce hydrogen. The pilot plant also includes equipment to prepare and feed char to the high-pressure reactor system, and to process and clean the hydrogen-rich product gas and the waste solids and gases. Supporting research to guide plant operations includes kinetic studies, evaluation of various chars for the process, and solids attrition and pneumatic lift-line studies.

**RECENT WORK AND ACCOMPLISHMENTS** — Construction of the pilot plant was completed in mid-1976, and start-up operations began shortly thereafter. Pilot plant operations during the early part of this year were directed toward start-up of the three main systems; the slurry vaporizer, the producer reactor, and the steam-iron reactor. Present operations are directed toward operation of the combined systems. The slurry vaporizer was the first system tested, and four slurry vaporization tests have been conducted over the past year. These operations have demonstrated the feasibility of feeding coal char at high pressure to the producer reactor. Five tests in the producer reactor have also been conducted. The first test was limited to char feeding and discharge. Subsequent tests were directed towards establishing and controlling bed heights in the producer, initiating gasification by injection of air and steam, and achieving self-sustained gasification at 1600<sup>o</sup>-1700<sup>o</sup>F. Six solids circulation tests in the steam-iron reactor have also been conducted. Refractory breakage caused flow interruptions in initial tests, and the solids life system had to be modified to solve this problem. After modification, solids circulation could be achieved for extended periods of time. The use of a new type of valve in the solids seal leg which feeds the upper reducer bed allowed smooth circulation over the entire operating range.

**PLANS FOR THE COMING YEAR** — Work on this project will be completed in FY 1978.

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\*Current contract only.



## PIPELINE GAS FROM COAL HYDROGENATION (HYDROGASIFICATION PROCESS)

INSTITUTE OF GAS TECHNOLOGY

DOE - \$36,720,000; American Gas Association - \$18,360,000

**OBJECTIVES** – This project intends to develop an economically attractive process for producing high-Btu pipeline gas from coal to supplement and extend natural gas supplies. Given the dwindling natural gas resources, substitute natural gas from coal could play a major role in meeting the nation's energy needs. The HYGAS process has the advantages of high efficiency, an ability to process all types of coal, and the ability to remove all potential pollutants from coal during the conversion process. The process operates at high pressure and the purified product gas can be directly introduced to the high-pressure gas transmission system. The pilot plant includes coal preparation and pretreatment sections, a slurry preparation section, and a gasification reactor consisting of a slurry dryer, two hydrogasification stages, and a steam-oxygen zone in which the hydrogen-rich gas needed in the hydrogasification stages is produced. The pilot plant also has gas purification and methanation sections which allow production of high-Btu gas of pipeline quality. Specific objectives in this plant since October 1976 have included the conclusion of a test series with Montana subbituminous coal, and the initiation of a test series with a mildly caking Illinois bituminous coal to test optimum operating conditions in the HYGAS reactor. This last test series has been aimed toward acquiring data at high coal conversion and high throughputs in order to develop a firm data base for a commercial/demonstration plant design based on the HYGAS process.

**RECENT WORK AND ACCOMPLISHMENTS** – Prior to October 1976, the feasibility of the HYGAS process was demonstrated on both lignite and bituminous coals. During the fall of 1976, four tests were conducted with Montana Rosebud seam subbituminous coal. Test 58, the most successful of these, extended over 11 days, during which all sections of the plant were activated. After this test, the Institute of Gas Technology was directed by the DOE/American Gas Association Operating Committee to study the HYGAS process with a mildly caking bituminous coal from the Illinois Basin. The purpose was to test conditions for optimizing the HYGAS process and, specifically, to determine conditions for maximum char conversion, high coal throughput, and minimum pretreatment conditions. Prior to these tests, modifications and improvements were made to the plant. Seven tests have been conducted in this test series during FY 1977. Operations at reasonable throughput rates and coal conversions have been established in this test series; however, additional pilot plant development work is being carried out to establish optimum economical conditions without the formation of clinkers.

**PLANS FOR THE COMING YEAR** – Tests with bituminous coal will continue, to determine operating conditions to develop a firm data base for a commercial/demonstration plant design.

## JOINT ENGINEERING SUPPORT FOR COAL GASIFICATION PROGRAM

C.F. BRAUN & CO

DOE - \$3,819,679; American Gas Association - \$1,909,839

12/1/75 - 1/31/78

**OBJECTIVES** – In general, the evaluation contractor for this program acts as a technical extension of the DOE Division of Coal Conversion and the Gas Research Institute for the joint



government-industry program for development of technology to convert coal to pipeline gas. Specifically, the evaluation contractor carries out evaluation activities in the areas of general program activities, factored estimates, process studies, and mechanical development. General program activities cover pilot plant surveillance, progress report evaluation, review and evaluation of pilot plant data, environmental coordination with other agencies, materials of construction program evaluation, and proposal evaluations. Factored estimates involve preparation of commercial concept designs and gas cost calculations for the processes in the program, using western and eastern coal feeds. Process studies involve all the unit operations conducted in converting coal to pipeline gas. The purpose of these studies is to select the best technology in each type of process operation for comparative economic evaluation of the processes in the joint program. Mechanical development involves identification of equipment that will be required in commercial-scale gasification plants that is not currently being manufactured because of the pioneer nature of the process or because the size or capacity exceeds that of similar items commercially manufactured. The second portion of the development involves investigation of the capability and interest of vendors of specific equipment items to undertake development contracts to produce the needed equipment.

**RECENT WORK AND ACCOMPLISHMENTS** — In the area of general program activities, pilot plant surveillance is a continuing activity as the pilot plants make successful runs and produce data for evaluation. Tests at both the HYGAS Steam-Oxygen and CO<sub>2</sub>-Acceptor pilot plants on lignite feed confirmed technical feasibility in accordance with criteria established to verify operability. Evaluation is continuing on other processes and with other feeds to establish the validity of design data produced during pilot plant operations. The materials evaluation program has progressed through most of its five phases, and preliminary materials selections for equipment have been described in an interim report submitted to DOE. A report on factored estimates for commercial designs using western coal has been published showing the projected gas costs for the processes under development. A similar report based on eastern coal is in progress. Process studies have been completed on many of the processing steps in coal gasification, and several others are still in progress. Programs are in progress to develop a vertical lift dryer for coal slurry, an instrument to measure the flow of coal solids in transport lines, and an instrument to measure the high temperatures existing inside gasification reactors.

**PLANS FOR THE COMING YEAR** — Work will include a continuation of pilot plant surveillance, completion of factored estimates on eastern coal and lignite, and completion of study reports still in progress. These include studies on acid gas removal, sulfur recovery, combined shift methanation processes, plant size, and the economics of recovering sulfuric acid versus elemental sulfur. Plans have been formulated to field test a solids flow meter, an instrument for measuring high temperature in a gasifier, and a vertical lift dryer. Mechanical development work will be concluded with publication of a guide covering the basic requirements for the design and construction of gasifier vessels.

## **SYNTHANE PROCESS — RESEARCH AND DEVELOPMENT**

### **PITTSBURGH ENERGY RESEARCH CENTER**

DOE - \$2,571,000

1955 - Continuing

**OBJECTIVES** — Technical support for the 72-ton/d Synthane pilot plant will be provided and a data base and improved processing steps will be developed which can be utilized in high-Btu

gasification processes, such as Synthane, and in co-product plants producing both SNG and liquid products. These steps include fluidized-bed gasification, coal feeding and pretreatment, catalytic methanation, catalytic synthesis of liquid products, tar recovery and utilization, and treatment of plant effluents to minimize environmental damage.

**RECENT WORK AND ACCOMPLISHMENTS** – Methanation reactor systems are being developed that feature low pressure drop and maximum recovery of the heat of reaction as high pressure steam. These reactors include the tube wall reactor, hot gas recycle reactor, and the hybrid reactor. A hybrid reactor utilizing eight-finned cast Raney nickel inserts was operated for more than 12 months. At the end of this operation, CO conversion was still in excess of 90 percent, and based on the rate of CO increase in the product gas along with changes in the catalyst temperature profiles, catalyst life may be extrapolated to 2.2 years. Analysis of the catalyst after reactor shut-down indicated a thin layer of nickel carbide on the outer surface of the catalyst with layers underneath still in active form. The carburizing was more extensive at the reactor inlet. A hot gas recycle test was initiated in a rebuilt reactor system using a gas ejector for recycle and an all-stainless steel system. This should help eliminate previous problems of catalyst fouling with compressor oil and with iron carbonyl formed from the carbon steel piping. A segmented reactor was tested as part of a cooperative agreement with the National Bureau of Standards. Various surface analysis techniques will be used to determine catalyst deactivation mechanisms. A continuous flow stirred tank reactor system was constructed and used to develop reaction kinetics for flame sprayed Raney nickel catalysts. A rate expression was determined which is being used to predict reactor performance in mathematical models of the various methanation reactors. The 40-atm fluidized-bed Synthane PDU gasifier was used in a series of parametric gasification tests to define the effect of coal throughput and pressure on product composition, yields, and conversion. A kinetic expression has been derived. A series of runs were made with Montana Rosebud coal to determine trace element balances around the gasifier. Analyses will be completed shortly. North Dakota lignite was also gasified in a series of tests to define the effect of particle size (and heat-up rate) on effluent production. Kinetic and equilibrium models of the gasifier have been developed. They are being used to analyze PDU and pilot plant data and will be useful for scale-up purposes. Tar deposition and ash agglomeration models have also been developed.

Many tests were performed in the 40- and 70-atm fluidized-bed pretreaters to develop start-up and shutdown procedures to be used in the 72-ton/d Synthane pilot plant. These procedures are being used in the current pilot plant tests with Illinois No. 6 coal. The entrained pretreatment unit was used to determine coal residence times in pneumatic transport to better define system kinetics. An atmospheric pressure two-dimensional cold model of the Synthane pilot plant gasifier was constructed and operated to determine the cause of clinker formation that has been encountered in several pilot plant runs. Dead spots were observed between fluidization jets which are believed to contribute to clinker formation. Several modifications were tested to determine the best method to be used in the pilot plant. Characterization and treatability of gasification wastewaters is also being studied. In particular, the gasification byproduct waters from the Synthane PDU have been studied. This research has concentrated on three major areas: pretreatment, dealing with the removal of oils, tars, and ammonia; biological treatment, both aerobic and anaerobic; and char sorption, which is use of Synthane byproduct char as an adsorbent for Synthane water contaminants. Preliminary measurement by atomic absorption analysis of twelve trace elements (Ni, V, Fe, Mg, Al, K, Ba, Zn, Mn, Cu, Co, and Cr) in the byproduct water has been made, with the untreated condensate meeting the proposed EPA criteria for public water supply for those elements. The biological kinetics of

both aerobic and anaerobic systems were studied using both diluted and undiluted condensates. A two-stage aerobic bio-system with diluted feed (24-hour detention time) gave 99.3 percent phenol reduction, 84.9 percent COD reduction, and 84.4 percent TOC removal over the first stage with little additional removal across the second stage. The second stage, however, acts as a buffer and tends to damp out fluctuations in effluent quality. Similar quality effluent from a bio-unit treating undiluted wastes requires a detention time of 5-6 days. Char sorption studies were made with raw condensate water to determine the removals of COD, TOC, phenols, organic acids, metals, cyanides and thiocyanates; the effect of pH on sorption and the biodegradability of char treated effluents.

**PLANS FOR THE COMING YEAR** — Support studies will continue, and in gasification, combined entrained pretreatment and deep-bed injection will be studied. Conceptual designs and economic assessments will be completed for the three methanation systems. An "extractive pretreatment" unit has been constructed and will be operated in the coming year. A high-pressure cold model will be constructed to observe fluidization under pressure. Wastewater characterization and treatability studies will continue.

### HYDROGASIFIER DEVELOPMENT

ROCKWELL INTERNATIONAL CORP./ROCKETDYNE DIVISION  
SUPPORTED BY CITIES SERVICE RESEARCH AND DEVELOPMENT CO.  
DOE - \$1,530,000  
2/77 - 8/78

**OBJECTIVES** — A single-state, short residence time reactor design for coal hydrogasification will be developed and the process data needed to design a pilot plant based upon that reactor design will be generated. The basic concept is to produce 90 percent or more of the product methane for SNG by direct reaction of coal with hydrogen. Therefore, the reactor's function is to accomplish rapid-rate pyrolysis of pulverized coal in the presence of hot hydrogen at elevated pressures. High volumetric throughputs are achieved, and short reactor residence times are controlled by using an entrained flow type reactor. Rapid and thorough mixing between reactant streams is accomplished by means of techniques adapted from very high throughput rocket injection technology. The coal particles are heated so rapidly near their injection point that they quickly become sufficiently devolatilized, and, by the time they encounter significant particle-to-particle contact or collision with the reactor wall, they are no longer prone to agglomerate. Thus, caking coals can be fed to this reactor without particle agglomeration or reactor plugging becoming serious problems.

**RECENT WORK AND ACCOMPLISHMENTS** — Bench-scale hydro-pyrolysis experiments have been carried out to define reactor pressure, temperature, and residence time conditions required to attain conversion of Montana subbituminous coal sufficient to support a balanced hydrogasification process. An engineering-scale (1/4 t/hr) reactor system has been constructed and activated, and is providing confirmatory data on a scale large enough to provide confidence in the proposed pilot plant reactor design. In addition to subbituminous coals, experiments are being made in this reactor with a mildly caking Illinois No. 6 hvCb coal and a highly caking Kentucky No. 9 hvAb coal. A computerized analytical model for a complete hydrogasification process has been formulated and programmed. It is being employed to perform detailed process flow studies in order to evaluate the many interrelated processing steps. The model includes factored economic correlations, so the pilot plant design can reflect features needed to optimize the cost of gas from a commercial scale (250 million scf/d pipeline SNG) gasification plant.

**PLANS FOR THE COMING YEAR** — Test durations in the 1/4-t/hr reactor will be extended from the current 8 min. to 1 to 8 hours. Conversion data obtained with the three coals will be correlated to a pilot plant design.

### DILUTE-PHASE HYDROGASIFICATION PROCESS (MODIFIED HYDRANE PROCESS)

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$800,000

1968 - Continuing

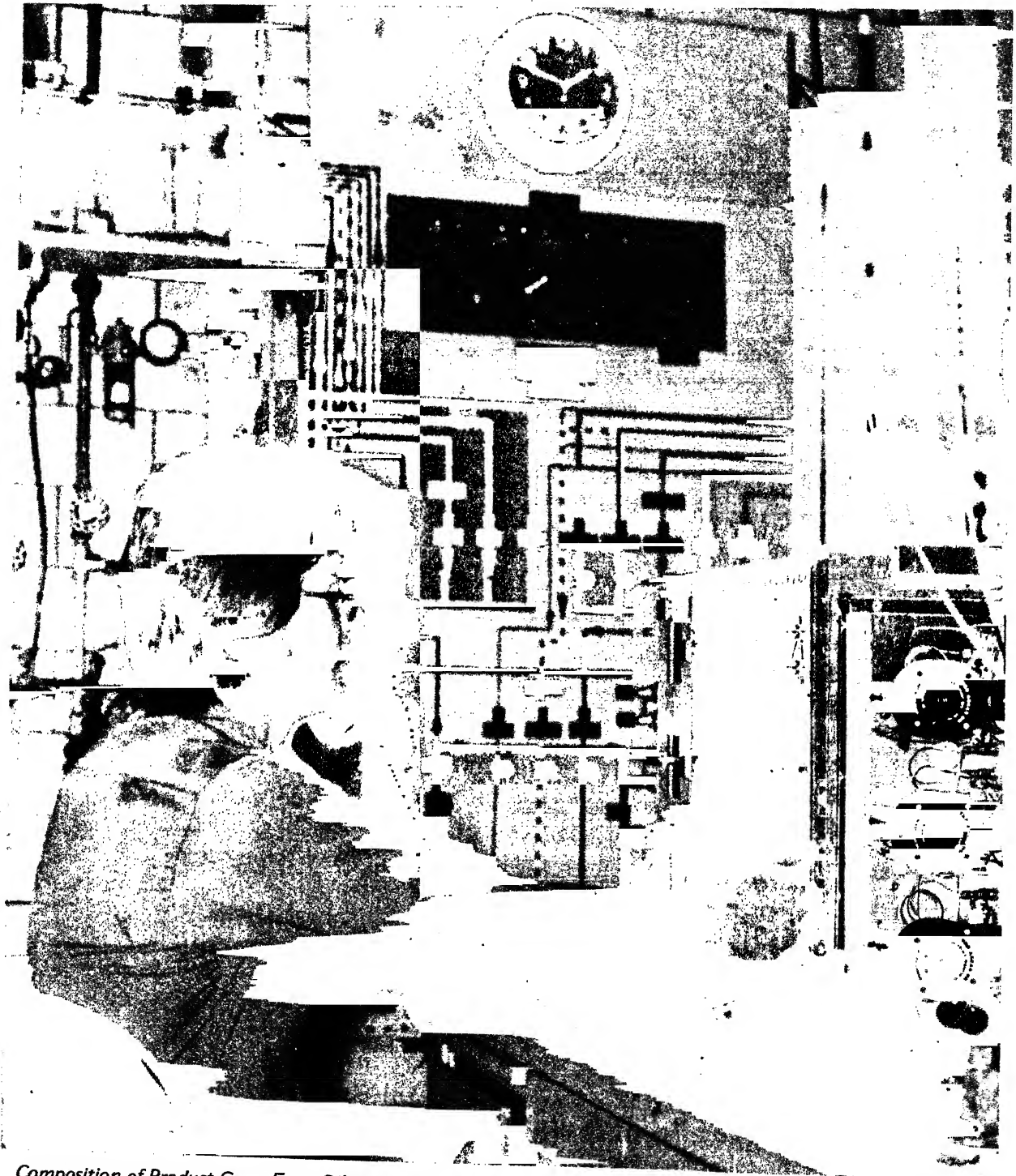
**OBJECTIVES** — This development project will demonstrate long-term operational reliability and acquire scale-up data for a third generation process which produces high-Btu substitute natural gas from coal by direct reaction with hydrogen in a dilute phase, free fall reactor. Primary attributes of this process include the simplicity of reactor mechanical design and process control. Also, 95 percent of the methane in the product gas is produced in the single-step reaction with hydrogen, thereby eliminating the large capital cost of equipment for secondary methanation of the intermediate syngas produced in two-step second-generation processes. Finally, higher energy conversion efficiency can be attained.

**RECENT WORK AND ACCOMPLISHMENTS** — Initial efforts during the year consisted of facility modifications to permit extended continuous operation. Major hardware additions were a dense phase, cold transport system for transfer of coal from ground level to the upper high-pressure feed lock hopper, a second high-pressure feed lock hopper, and a parallel liquid-byproduct trap system. The 5-ft by 3-in ID heated reactor was replaced by a 9-ft unit with the capability of providing various heated lengths of 1-9 ft in 2-ft intervals. Using the new hardware, a continuous hydrogasification experiment of 45-hours duration was completed. Several runs were made with lignite at reaction conditions of 900°C and 1000 psi to expand the range of available kinetic and conversion data. Carbon conversion of 51 percent, as needed for process balance, was achieved at the preferred low hydrogen-to-coal ratios. The product gas has a heating value of 1000 Btu/scf after cleanup and light methanation to remove minor quantities of CO (10 vol percent). A series of experiments to determine the effect of coal feed rate showed that tripling the throughput to 800 lb/hr ft<sup>2</sup> reduced the volume of methane in the raw product gas by only 8 percent. Installation of the dilute phase reactor cold model facility (10 ton/d capacity) was completed and shakedown tests initiated. It will be used to develop coal and gas injection methods, flow distribution systems, and to define particle velocity and residence time in the free fall reactor. Fast photographic techniques are being developed for particle velocity measurements using char from Illinois No. 6 coal.

A real-time process data acquisition system was installed, and the on-line gas analysis system was upgraded to include analyses for C<sub>2</sub>–C<sub>8</sub> and H<sub>2</sub>S and to provide rapid evaluation of the experimental results.

**PLANS FOR THE COMING YEAR** — Emphasis in the near future will be placed on the successful completion of several experiments of 200-hours duration to demonstrate long-term operational reliability. Optimum conditions for processing lignite at high throughputs (800 lb/hr ft<sup>2</sup>) will be defined. Testing with lignite will be completed and all process data analyzed, correlated, and arranged in a suitable format to provide the baseline information necessary for engineering design of a 10-30 ton/d PDU. Testing to acquire process scale-up data for subbituminous and bituminous

coals will be initiated. Also, shakedown tests of the cold model will be completed and studies of the dynamics of gas-particle interaction during free fall will be initiated to support design of a more efficient reactor configuration.



*Composition of Product Gases From Dilute-Phase Hydrogasification Experiments Are Monitored by Means of an On-Line Gas Chromatograph*

## HYDRANE PROCESS SCALEUP

MORGANTOWN ENERGY RESEARCH CENTER  
DOE - \$50,000  
7/74 - Continuing

**OBJECTIVES** – The Hydrane process produces methane from coal by direct hydrogenation. Little catalytic methanation is required because 99 percent of the product methane is produced directly in the reactor. This process, initiated at PERC in 1968, is being studied for feasibility of scaleup, to a plant size, that would give useful information for further assessment toward the design of a PDU and a commercial-size plant.

**RECENT WORK AND ACCOMPLISHMENTS** – The Hydrane program was redirected in June 1976 toward modifying the process to use a simple single-stage reactor. Although the two-stage design was determined feasible by an independent study, a less complicated reactor is expected to reduce operating problems. The single-stage concept is being developed jointly by PERC, MERC, and several DOE contractors. Rocketdyne was contracted to develop a high throughput hydrogasifier. Cities Service is doing laboratory-scale work on the process as a subcontractor to Rocketdyne. Bechtel was contracted for a feasibility study of the process. PERC developed the original Hydrane concept and is continuing to develop their Dilute Phase Hydrogasification reactor. Operation with lignite is being tried first because of its higher reactivity. Coal with high reactivity is required to obtain the desired 50 percent (or more) conversion in a single-stage reactor. Dravo, under contract to MERC, is determining the impact of the single-stage concept on their previous feasibility study of the two-stage reactor. Preliminary work by Dravo indicated that sufficient data were not available to do a scaled-up single-stage reactor design. Their contract therefore was extended to await data from PERC and Rocketdyne. They were further authorized to study and recommend a concept based on cryogenic separation, for cleaning the hydrogasifier output. A report on this concept is scheduled in December 1977.

**PLANS FOR THE COMING YEAR** – Dravo will furnish their conceptual design for a single-stage reactor based on data furnished by Rocketdyne and PERC. The DRAVO gas cleanup concept report will be completed when adequate response is obtained from vendors of cryogenic equipment. MERC will monitor and provide technical support to the program.

## IMPROVED FORMS OF CATALYTIC MATERIALS FOR METHANATION

DEPARTMENT OF THE INTERIOR, ALBANY METALLURGY RESEARCH CENTER  
DOE - \$1,350,000  
7/74 - Continuing

**OBJECTIVES** – Cast Raney nickel inserts will be provided for large-scale testing at Pittsburgh Energy Research Center. To increase both the resistance to poisons and the methanation activity of the basic Raney nickel formula, alloying and heat treating will be performed. Specific objectives have been to supply all of PERC's needs for cast finned inserts, supply cast thin catalyst plates, 500 ft of flame spray rod, and various catalyst shapes for laboratory scale reactors. Other objectives are to prepare powdered catalyst alloy and cast catalyst shapes of various compositions. These efforts support the Synthane program for converting coal to substitute natural gas.



**RECENT WORK AND ACCOMPLISHMENTS** – Finned cast massive catalysts functioned efficiently for one year, far surpassing other catalysts tested. Over 500 ft of rod made from aluminum-encapsulated Raney nickel powder were prepared. The rod will be used to flame spray catalyst onto a suitable surface. Other catalyst shapes prepared for PERC were thin Raney nickel plates and various catalytic shapes for use in bench-scale methanators. Knowledge gained in casting techniques will be used to show commercial foundries how to make castings of this material. In addition to providing hardware, metallographic studies and analyses have been made of the materials produced. New contributions to Raney nickel metallurgy have been made. The Bureau recently constructed a small methanation reactor containing two 1/2-inch-diameter reactors to correlate metallurgical and crystallographic structures with catalytic activity. The apparatus is equipped with a gas chromatograph and other instrumentation for studying catalytic poisoning, in situ regeneration, and the effect of metallic impurities in Raney nickel. Some initial studies, coupled with thermodynamic calculations, have shown that poisoned Raney nickel cannot be regenerated, so it is important to develop sulfur resistant catalysts. Research on determining the effect of additives in a Raney nickel catalyst showed that the addition of calcium, carbon, or molybdenum improves the catalytic activity.

**PLANS FOR THE COMING YEAR** – In FY 1978, the Bureau of Mines will prepare cast catalyst shapes for use in tests at the Pittsburgh Energy Research Center, and evaluate modified Raney nickel catalysts for life, high-temperature stability, and other physical, chemical, and metallurgical factors which could influence the operation of the methanation step. Specific hardware requests to be completed include catalytic test samples containing various promoters, finned massive inserts containing promoters, promoted alloy powders, and equilibrated inserts. Raney nickel powder will be encapsulated in aluminum and swaged to small diameter flame spray rod.

## COAL GASIFICATION USING CHEMICALLY INCORPORATED CALCIUM OXIDE

BATTELLE, COLUMBUS LABORATORIES

Battelle - \$941,571

4/76 - 5/77 - 9/77 - 2/78

**OBJECTIVES** – This program seeks to increase the reactivity and eliminate the swelling and caking properties of eastern bituminous coals by chemically incorporating CaO into the coal. The treatment process could, in concept, be carried out as part of a conventional slurry feeding system requiring only the addition of a reactor vessel to provide the residence time necessary to chemically react the CaO with the coal. One of the major problems in the utilization of eastern coals is their swelling and agglomeration properties in gasifier environments and their low gasification reactivity (compared to western coal) which lowers reactor throughput and increases steam and oxygen requirements. The catalyzation process investigated may allow these problems to be overcome or reduced significantly, thus facilitating the use of eastern coals in gasification processes.

**RECENT WORK AND ACCOMPLISHMENTS** – Illinois No. 6 coal was treated at conditions at which it is known to convert to the equivalent of a lignite in regard to both swelling and caking properties and reactivity. This treated coal was used as a feedstock for direct hydrogasification (at Battelle Columbus Laboratories) and some preliminary steam/oxygen gasification tests were made at PERC in a 4-inch Synthane reactor. The PERC preliminary tests indicated that  $H_2 + CO + CH_4$  yields for the catalyzed coal were increased by factors ranging from 1.38 to 2.14 (depending on temperature) compared to the raw coal, which was oxidized before being fed to the reactor to avoid

swelling and agglomeration problems. Direct hydrogasification tests in a single-stage reactor demonstrated that SNG could be produced from the product gas without hydrogen separation over a relatively wide range of operating pressures.

**PLANS FOR THE COMING YEAR** — The benefits of using the catalyzed coal in a steam/oxygen gasifier will continue to be assessed by PERC. The direct hydrogasification effort will also continue with emphasis on two-stage hydrogasifier operation. In addition, simple modifications of the basic catalyzation system will be made to enhance hydrogasification reactivity (the basic process enhances steam gasification reactivity but does not have a significant effect on hydrogasification reactivity).

### LIQUID PHASE METHANATION PILOT PLANT OPERATION AND LABORATORY SUPPORT WORK

CHEM SYSTEMS, INC.  
DOE - \$601,700  
7/76 - Continuing

**OBJECTIVES** — A practical and useful process will be developed for converting coal-derived synthesis gases (largely carbon monoxide, hydrogen, and methane) into methane using liquid fluidized beds. The successful completion of this process development will provide a simple and economical process for upgrading low-Btu synthesis gas to high-Btu substitute natural gas (SNG) which will be applicable to all coal gasification processes. The liquid phase methanation (LPM) process can be used as the final conversion step with any of the coal gasification processes presently under development by DOE. The process should also offer a high operating reliability due to its ability to smooth out upsets in synthesis gas feed rates, composition, and temperature. Furthermore, since essentially complete carbon monoxide conversion to methane can be achieved in a single pass, the need for rotating equipment to recycle unconverted synthesis gas is eliminated. This allows a high thermal efficiency for the process and may result in SNG costs \$0.15-0.25/MMBtu lower than those obtained using presently offered technology. The LPM pilot plant constructed in accordance with the requirements of a prior contract is to be test operated on the synthesis gas produced by the Institute of Gas Technology's HYGAS pilot plant. After completing the experimental program at that site, the LPM pilot plant will be disassembled and shipped to a second coal gasification pilot plant site. In addition to pilot plant test runs, laboratory support work will be conducted whenever necessary to supplement pilot plant operations. This includes testing of liquids and catalysts that would be suitable for pilot plant operation. An experimental program will be conducted on the rates of carbon formation in methanators.

**RECENT WORK AND ACCOMPLISHMENTS** — The component sections of the skid-mounted LPM pilot plant arrived in Chicago in November 1976. The unit was reassembled, installation was completed including several modifications, and shakedown was completed during one of the coldest winters in history. Between March and October of 1977, four separate test runs were accomplished. The LPM pilot plant logged a total operating time in excess of 1300 hours including 204 hours when synthesis gas was being methanated. The four runs included testing two different catalysts and two different feed gas streams (HYGAS synthesis gas and steam-methane reformer gas). Run No. 1 took place in early April and included a total of 27 hours of accumulated methanation time. The LPM unit operated for 3 hours on synthesis gas from the HYGAS pilot plant, and 24 hours on synthesis gas from IGT's catalytic steam-methane reformer. Process variable scans were performed



during the 24-hour period. Calsicat Ni-230S was the catalyst. Run No. 2 was performed during May with a new charge of Calsicat Ni-230S catalyst. Steam-methane reformer gas was fed to the unit with an  $H_2/CO$  ratio of approximately 6.5. The LPM plant was run for a total of 60 hours during which time several different process conditions were tested. Run No. 3 took place during August with a catalyst supplied by the International Nickel Co. The run lasted for 70 hours feeding synthesis gas from the HYGAS reactor. Reactor temperature of 600 to 675°F and pressures of 500 and 750 psig were tested. CO conversions ranged from 46 to 96 percent depending upon synthesis gas flow rates and reactor conditions. Run No. 4 took place during September with the same catalyst as used in Run No. 3. Synthesis gas from the IGT's steam-methane reformer was fed to the unit, and the run lasted 46 hours.

Several achievements were made as a result of these operations: (1) The basic operability of the process was demonstrated in a 50-fold scale-up from a smaller process development unit, (2) process variable scans were performed and indicated that catalytic behavior in regard to temperature and gas flow rates is as predicted from previous work, and (3) the liquid phase reaction system was found to be impervious to the massive sulfur contamination which occurred in Run No. 1 during an upset in IGT's acid gas removal system.

**PLANS FOR THE COMING YEAR** — The pilot plant operations to date have identified a problem in gas distribution within the reactor. Plans for the near future will concentrate on improving gas distribution in the reactor. Two or more different catalyst support plates and distribution/sparger systems will be designed and tested. Additional process variable scans will be performed to complete the experimental problem at HYGAS. A short-term continuous run will be made to monitor catalyst activity. Upon completion of the program, the LPM pilot plant will be shut down, disassembled, and shipped to a second coal gasification pilot plant site. An experimental program will be performed at the second site using synthesis gas generated from the coal gasifier. Laboratory support work will be performed during pilot plant operations as required to test new catalyst candidates for pilot plant operation and help solve any operating problems that may arise. The experimental test units for the rates of carbon formation program will be completed and test runs will begin.

## **DEVELOPMENT OF PEAT GASIFICATION**

MINNESOTA GAS COMPANY  
DOE - \$1,037,311; Minnesota Gas - \$203,426  
7/1/76 - 6/30/78

**OBJECTIVES** — The program will experimentally obtain the necessary peat gasification data in 'DU scale equipment. Based on experimental results, a peat gasification technical

**RECENT WORK AND ACCOMPLISHMENTS** – The laboratory-scale tests have been completed and show that peat is more reactive than lignite; peat should require a smaller gasifier, lower operating pressures, a smaller final catalytic methanator, and a less costly gas purification section than is required for lignite. Process development unit tests are underway on peat hydrogasification and peat char gasification to confirm laboratory data and determine effects of initial scaleup.

**PLANS FOR THE COMING YEAR** – Work will include completion of process development tests on peat hydrogasification and peat char gasification, evaluation of laboratory and process development unit data, and economic analysis of the peat hydrogasification system.

## SCALEUP REQUIREMENTS OF CATALYTIC COAL GASIFICATION PROCESS

EXXON RESEARCH AND ENGINEERING COMPANY

DOE - \$1,018,820

11/1/76 - 3/31/78

**OBJECTIVES** – The scaleup needs of the Exxon catalytic coal gasification process are being defined and the feasibility and cost of adapting an existing DOE-sponsored coal gasification large pilot plant is being evaluated. The principal tasks are to: (1) develop a study design of a conceptual grass-roots large pilot plant for the catalytic gasification process and estimate the construction and operating costs, (2) study the feasibility of using one of three existing pilot plants (Hygas, Steam-Iron, or Synthane) for catalytic gasification scaleup and determine which is most suitable, and (3) complete a study design and estimate of construction and operating costs for a revamp of the selected pilot plant.

**RECENT WORK AND ACCOMPLISHMENTS** – The conceptual study design for the grass-roots large pilot plant has been completed. The coal feed rate (92-t/d) was set by the minimum reactor size that is needed to provide fluid-bed scaleup data that can be used for design of a pioneer commercial plant with acceptable risk. Facilities were included so that all key process steps could be operated in an integrated manner. Construction costs for the grass-roots pilot plant were estimated to be \$130,000,000 for a U.S. Gulf Coast location. Adjusting the grass-roots case cost estimate for Pittsburgh area wage rates and productivity results in an estimate of \$150,000,000. Projected mechanical completion is 4Q 1982 for a project entering the basic design phase in 4Q 1979 and awarding detailed engineering and construction contracts in 2Q 1980. Operating costs total \$50,000,000 to \$60,070,000 for a 30-month testing period running from 4Q 1982 into 2Q 1985. The Synthane pilot plant was selected as best suited for retrofitting to develop the catalytic gasification process. Principal reasons for selection of the Synthane unit are the greater land availability at the site and the larger number of process units that can be used directly or modified in a revamp. The study design and cost estimate for the Synthane revamp case were completed.

**PLANS FOR THE COMING YEAR** – Alternative pilot plant strategies that could speed the development of the catalytic gasification process will be evaluated. One promising approach is to carry out a minimum conversion of the existing Synthane unit to allow for early operation of the gasification section in the catalytic mode. Facilities for catalyst recovery and recycle and synthesis gas recycle could be added in later stages. The cost, schedule, and technical merits of this approach will be studied under a no-cost contract extension.

## COAL BENEFICIATION FOR COAL CONVERSION/UTILIZATION

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$100,000

1977 - Continuing

**OBJECTIVES** — It will be experimentally determined whether coal beneficiation to remove mineral matter, including inorganic sulfur compounds, has beneficial or adverse effects on coal conversion to liquids or gases and on fluidized-bed combustion. To develop specifications for advanced coal utilization feedstocks for optimal processing, data are being gathered to assess whether particular coals, prior to their use in a conversion or advanced utilization system, should be cleaned as part of the optimum plant design. The technological advantages and disadvantages of using coal beneficiation as an initial treatment must be evaluated to determine its potential through decreased technical difficulties in coal processing system operation and increased economic benefits in coal conversion/utilization.

**RECENT WORK AND ACCOMPLISHMENTS** — Forty tons of a high volatile bituminous coal has been obtained as the first of several types planned for experimental study. A portion of the as-mined coal was physically cleaned to achieve a substantial reduction of inorganic sulfur and other mineral matter using commercially applied procedures. Portions of the as-mined coal, the clean product from coal beneficiation, and the cleaning plant reject material were custom-sized for use in coal liquefaction, coal gasification, and fluidized-bed combustion tests. Detailed physical and chemical characterizations of the raw coal, cleaned product, and reject material are underway. Program schedules have been developed for testing of the coal fractions in gasification, liquefaction, and fluidized-bed combustion operations. To support these tests in continuous process units, batch studies have been initiated. Orientation runs have been completed to assess the parameters for conducting batch studies, and more detailed batch runs are planned.

**PLANS FOR THE COMING YEAR** — For the high volatile bituminous coal, continuous experiments will be conducted for liquefaction and gasification of both the as-mined and cleaned coals and for fluidized-bed combustion of the as-mined, cleaned, and reject fractions. These continuous runs will be supported by additional batch studies. Detailed material and process characterizations will be performed for all these operations. Extensive chemical analyses, including studies of sulfur and trace element dispositions, product yields and qualities, and effluent characteristics, will be performed. Coal processing technology impacts and energy efficiencies will be studied. Engineering design studies will be initiated to evaluate the technical and economic benefits/drawbacks of combined beneficiation and coal conversion/utilization. A second coal of subbituminous rank will be selected for study. A large tonnage quantity will be obtained, cleaned, sized, and processed for determination of the impact of beneficiation.

## COAL HYDROGASIFICATION PROCESSES

BECHTEL CORPORATION

DOE - \$170,200

2/1/77 - 11/30/77

**OBJECTIVES** — An analytical study will be conducted to investigate the operability potential and scaleup feasibility of the Cities Service, Rocketdyne, and Pittsburgh Energy Research Center (PERC) coal hydrogasification processes, relative to DOE plans for a hydrogasification process

development unit (PDU). As part of the program objective, a reactor model study is being performed for each of the three processes, and a conceptual, full-scale hydrogasification reactor design is being generated. The reactor systems operate at temperatures up to 2000°F and pressures to 3000 psi. Reactor product is primarily methane, with smaller amounts of ethane, BTX, light oils, and carbon-oxides. Hydrogasification offers a potential for economic and efficient production of synthetic pipeline gas from coal, to supplant rapidly dwindling natural gas reserves.

**RECENT WORK AND ACCOMPLISHMENTS** – Data from Rocketdyne tests using subbituminous and bituminous coal, from Cities Service tests using lignite and subbituminous coal, and from PERC tests using various rank coals have been entered into a computerized data base. Semi-empirical correlations for predicting carbon conversion efficiency and carbon selectivity to gaseous and liquid products have been fitted to the data. The fitted models indicate that the Cities Service and Rocketdyne reactors behave similarly for the same coal. A conceptual design of a full-scale hydrogasification reactor has been generated, based on data gathered in the Cities Service and Rocketdyne reactors, together with the predictive reactor performance models fitted to the data. As presently visualized, a full-scale reactor facility would consist of a hydrogasification stage to produce methane-rich product gas from the coal, and a steam/oxygen gasification stage to produce hydrogen-rich product gas from the unreacted char.

**PLANS FOR THE COMING YEAR** – Data analysis will be completed and final report will be prepared.

## INSTRUMENTATION AND PROCESS CONTROL FOR FOSSIL DEMONSTRATION PLANTS

ARGONNE NATIONAL LABORATORY

DOE - \$1,000,000

7/1/76 - Continuing

**OBJECTIVES** – This project aims to assess the commercial availability of, or to develop, process control instrumentation for demonstration-scale coal gasification, liquefaction, and fluidized-bed combustion systems. Areas where commercial instruments are currently unavailable or inadequate for conditions found in coal plants include solids mass-flow, on-line analysis, level, and temperature. Wherever possible, current commercial instruments will be upgraded. Cooperation with industry and with pilot/demonstration plants will be maintained to expedite availability of adequate instrumentation. Reliable automatic process control is essential to the efficient, continuous, and safe operation of large-scale coal gasification, liquefaction, and fluidized-bed combustion systems. Instruments capable of monitoring process variables under the hostile conditions found in coal conversion and combustion processes must be available to achieve automatic control.

**RECENT WORK AND ACCOMPLISHMENTS** – Work continued on development and evaluation of mass-flow instruments for solids/fluids flow streams. A solids/gas flow test facility is near completion for mass-flow instrument testing and calibration. Work progressed on several mass-flow instruments using different physical techniques during FY 1977: an acoustic flow/no-flow instrument was completed for BIGAS, an acoustic flow test rig was completed for HYGAS, a capacitive flow test rig for HYGAS was nearly completed, fabrication of a gamma-ray transmission correlation test rig for HYGAS was begun, an acoustic flowmeter feasibility study was completed, and an industrial contract was awarded for an optical in-stream particulate monitoring instrument. Development of other on-line process control instruments continued: on-line composition

monitoring of coal by neutron-induced gammas was shown to be feasible, and a commercial capacitive level gauge for an oil/water interface was purchased for tests at HYGAS.

Process control analysis to establish control criteria and quantify instrument needs continued: instrument requirements for coal conversion plants can now be quantified using a program developed at ANL and tested at another site; and computer encoding of a model of the Ralph M. Parsons (RMP) methanation process was completed and readied for testing. Information exchange has involved organization of the first Symposium on Instrumentation and Control for Fossil Demonstration Plants held in Chicago in July 1977 with about 200 attendees from government, industry, national laboratories, and universities. Also, current information was collected and the draft nearly completed for an update of the state-of-the-art study on instrumentation for process control and safety in large-scale coal gasification, liquefaction, and fluidized-bed combustion systems. Furthermore, arrangements were made for publication and distribution of a quarterly newsletter concerning instrumentation in coal conversion and advanced combustion plants.

**PLANS FOR THE COMING YEAR** – During FY 1978, testing and analysis of instruments developed at ANL and elsewhere will take place at the ANL flow test facility and the BIGAS and HYGAS pilot plants: the acoustic flow/no-flow instrument will be tested at BIGAS; the acoustic, capacitive, and gamma-ray correlation mass-flow devices and the capacitive level device will be tested at HYGAS; and developmental tests of (ANL and other) flow instruments will commence on the loop facility. Development contracts will be awarded for correlation and capacitive devices using principles proven in the above tests. Research will continue on the on-line composition analyzers. Two industrial participants will work at ANL in acoustic and capacitive instrument development. A contract will be awarded for an acoustic temperature device. Mathematical analysis will continue on specified demonstration plant processes following application of the simulation program to the RMP methanation process. Information exchange will be continued with the ANL and DOE sponsored 1978 Symposium on Instrumentation and Control for Fossil Demonstration Plants being held in California in June. The first annual (1977) symposium proceedings will be distributed in FY 1978. The updated state-of-the-art report will be released in early 1978. The quarterly newsletter will begin in December 1977.

## **PROCESS MODELING SUPPORT FOR COAL CONVERSION PROCESSES**

**OAK RIDGE NATIONAL LABORATORY**

**DOE - \$150,000**

**7/1/76 - Continuing**

**OBJECTIVES** – Purdue University, Lehigh University, and Oak Ridge National Laboratory are working together to develop computer programs that can be used for the design, optimization, cost estimation, and economic evaluation of coal conversion plants. These computer programs will

to a partitioned data set to facilitate separation of the various source programs, subroutines, and data files. Work is in progress to eliminate incompatibilities between the Purdue CDC coding and the ORNL IBM-360 system. The ORNL computer program PRP for produce price calculation was expanded and modified. PRP is a discounted cash flow program for calculating the manufacturing cost of the product. A report has been issued that describes the basis for the program. Work continued on another ORNL computer program, PPL, which calculates piping layout, determines pipe diameters and lengths, and makes an overall piping cost estimate for a process plant. The purpose of the program is to provide a more accurate method for estimating the cost of process piping. The PPL program is currently operational but requires further refinement of various material and labor cost data. Work is continuing on the development of a computer program (HDC) that calculates the mechanical design of a shell-and-tube heat exchanger and estimates its costs. The HDC program uses the results of the thermal design as a starting point and calculates the mechanical design, the weights of all parts, and the estimated cost. The ORNL cost estimating program, CAPITAL, was copied and sent to Purdue. This program uses the factor method to estimate the capital investment for complete process facilities based on major process equipment costs. Further development of the program will be handled jointly by Purdue and ORNL.

**PLANS FOR THE COMING YEAR** – Following the completion of the Purdue and Lehigh simulators, Oak Ridge National Laboratory will provide computer facilities and personnel for the use of the simulators, in support of DOE-designated design and evaluation work. Oak Ridge National Laboratory will also train other DOE-designated contractors in the use of the programs.

#### COAL CONVERSION PROCESSES USING REFERENCE SIMULATOR

PURDUE RESEARCH FOUNDATION  
DOE - \$428,331  
3/76 - 5/79

**OBJECTIVES** – This project aims to construct a modular computer simulation/design package for coal conversion systems and to use this package to study in a programmatic fashion an array of coal conversion flowsheet alternatives. The computerized package is to be based on bench- and large-scale pilot plant data developed by other organizations under DOE contracts. It is to have sufficient flexibility to permit the user to incorporate process alternatives and engineering design modifications and is to have the capability for detailed cost estimation and economic evaluation. A process similar to the Illinois Coal Gasification Group (ICGG) Demonstration Plant based on COED and COGAS development work is the first conversion process to be investigated with the simulation package. The specific models developed for the base case ICGG application will be used in broader range design studies but in addition can be used to indicate promising process modifications and refinements as well as to assess the need for and importance of new process information and developments which may arise over the life of that demonstration project. Thus, the modified ICGG plant model will be of direct utility throughout the preliminary design, the final design, the operation, and the commercialization phases of this promising coal conversion process. The modification of the ICGG plant model has been necessary because certain process data in the plant flowsheet have been classified as proprietary by the ICGG contractors. Accordingly, substitutions have been made from data already published to assure that the plant model when complete may be published with no restrictions.

**RECENT WORK AND ACCOMPLISHMENTS** – A physical properties estimation package incorporating data limited to pure component species of interest in conversion plants has been encoded. A very efficient and novel large-scale material balancing program has been implemented and tested. A parallel energy balancing code is currently undergoing testing. A process equipment calculation system employing sparse stream and equipment vector storage and retrieval schemes is operational, and a substantial number of unit operation calculations modules have been incorporated within this system. Cost data for various process equipment types have been gathered, and data files have been implemented on the computer. Work is in progress on file manipulation and equipment costing routines which employ these files. A detailed process design model for a COED type multistage fluidized-bed coal pyrolysis unit has been developed based on COED pilot plant and literature kinetic data. Semi-empirical models have been assembled for the COED oil hydrotreating reactors. Macroscopic models of char gasification and synthesis gas methanation reactors suitable for preliminary design studies have been developed. Finally, a conceptual design study has been carried out to define a process flowsheet similar to the ICGG process. The modified COED process flowsheet developed differs from the ICGG proposal in that proprietary or otherwise ill-defined processing steps are replaced with units whose operating data are adequately reported in the open literature and for which satisfactory design models can be formulated.

**PLANS FOR THE COMING YEAR** – Within the next year the remaining portions of the simulation/design package will be completed. Areas which require further work include (1) the equipment costing and economic evaluation program library, (2) extension of the physical properties package to accommodate mixtures of pseudo-components, and (3) integration of the individual program package sections to permit a complete uninterrupted cycle of calculations from flowsheet balance calculations through economic evaluation. The complete base case model will be assembled for the modified COED process. This model will then be used in two types of case studies. The first type will seek to define the limits of feasible operating conditions and to assess their effects on process economics. The second type of case studies will examine the impact of alternative combustor/gasifier configurations different from those proposed by ICGG upon the overall process design and economics of the ICGG process. These studies will require the development of design models for these combustor/gasifier alternatives as well as for several additional process units affected by the selection of the former units.

## **SOFTWARE SYSTEM FOR SIMULATION OF COAL CONVERSION PLANTS**

**LEHIGH UNIVERSITY**  
DOE - \$351,030  
6/76 - 5/79

**OBJECTIVES** – Numerical algorithms, implemented as computer codes, will be developed for the

purpose of keeping excursions from desired steady state conditions within bounds. The effectiveness of proposed control systems can be studied only through dynamic simulation.

**RECENT WORK AND ACCOMPLISHMENTS** — The basic Differential Systems Simulator, Version 2 (DSS/2) for ordinary differential equations and partial differential equations has been completed and released to DOE at Oak Ridge National Laboratory and the Energy Support Group at Aberdeen Proving Ground along with complete sets of manuals and test problems. The variable-order, variable-step size integration algorithm of C.W. Gear (as implemented by A.C. Hindmarsh) has been installed as the first-line temporal integrator of the DSS/2 dynamic simulation code. This algorithm is implicit for stiff systems, and the Hindmarsh implementation is based on a banded specification of the ordinary differential equation system Jacobian matrix. Currently, only the diagonal approximation has been made available in the released versions of DSS/2. The basic system also contains 14 Runge-Kutta algorithms for ordinary differential equations. The partial differential equation capability is based on five central differencing subroutines, all easily available to the user. In addition, a series of utility routines for plotting and function generation enhance the system's convenience. DSS/2 has been distributed to other DOE contractors to test the transportability of the code. At all of the installations, representing a variety of computers, the code compiled successfully and executed the test problems without difficulty, thus giving every indication that DSS/2 is a transportable code.

The physical properties package developed by the DOE group at Purdue University has been installed on the Lehigh CDC 6400 for use within the DSS/2 system. This package consists of a data base and a set of subroutines which compute physical and chemical properties. The subroutines associated with single-phase properties have been modified, tested, and made available on-line for use in the simulation of plant units. The main part of the bulk methanation section of the plant, which consists of six fixed-bed catalytic adiabatic reactors in series between which and into which the temperatures were independently set to the values stated in the process description, has been simulated. Water-gas shift and methanation occur simultaneously over the same catalyst. One of the initial conditions considered was a constant-temperature profile in each reactor and a zero conversion profile in all reactors. The simulation was then begun by starting a reactant feed to the first reactor. Results from the temperature profile in each sector show that the dynamic simulation is approaching the steady state solution, generated from an independent calculation, as expected.

**PLANS FOR THE COMING YEAR** — Some additional refinements of the DSS/2 code are contemplated, particularly in response to user experience, but the major effort for the coming year will be the application of the DSS/2 system to the dynamic simulation of the individual units of a coal gasification plant. The two-phase sections of the physical properties package will be modified and tested for installation for on-line use in simulation of units of the plant. The methanation section of the plant will be expanded to include other components on the flowsheet and will be studied under several different transient conditions. The gasification section of the plant will receive attention next, followed by the pyrolysis section with high interest resting in linking two major sections of the plant together.



## PORTFOLIO MODEL

ECONERGY, INC.  
DOE - \$77,618  
9/14/76 - 3/31/77

**OBJECTIVES** – Criteria for evaluation and selection of a set of proposed coal conversion processes will be established. By incorporating the fundamental principles of portfolio theory, both the risks and the economic benefits, i.e., revenues less costs of capital, operation, and time, can be determined for a set of processes. The trade-off between benefits and risks for each possible set of coal conversion processes is illustrated by examining their relative positions on a benefit-risk map. Other objectives are to provide support to DOE management in terms of developing approaches to costing of coal conversion products as well as to joint funding participation by government and industry in coal conversion projects.

**RECENT WORK AND ACCOMPLISHMENTS** – The Econergy portfolio model has been developed and can be used for evaluating coal conversion processes as candidates for demonstration plant funding. The evaluation procedure utilizes information on process economics and process risks. Both kinds of information are combined for each process so that a comprehensive comparison among individual processes can be made. In addition, information regarding process similarities and dissimilarities is used to provide a comparison of various combinations of the processes. This approach to process evaluation is termed a portfolio approach because it allows the interrelated economic and risk implications of a group or portfolio of processes to be considered. Other accomplishments include three studies of economic and financial aspects of the coal conversion program. An examination of the assumptions underlying the utility method for determining a cost-based price of energy was made which illustrated the fact that current inconsistencies in these assumptions result in energy costs which can vary by as much as a factor of two. A cost sharing rationale, which provides a mechanism for appropriate sharing of project risk as well as cost between DOE and individual industry partners, was developed for government-industry investment participation in coal-based energy projects.

**PLANS FOR THE COMING YEAR** – Current work includes refinement of technologic risk assessments, incorporation of current cost estimates, treatment of differential inflation, and implementation of capital budgeting in the portfolio model. In addition, the cost and risk sharing rationale described above will be developed into a workable model for evaluating potential government-industry financial arrangements.

## WATER TREATMENT IN DEMONSTRATION PLANTS

WATER PURIFICATION ASSOCIATES  
DOE - \$263,000  
3/10/77 - 9/9/78

**OBJECTIVES** – Plants converting coal to other fuels can consume large amounts of water, both as a source of hydrogen and to be evaporated for cooling. Furthermore, most of these plants take in water of good quality for treatment to boiler feed or for use as circulating cooling water and put out highly contaminated water, which condenses out of process streams. To avoid pollution and to minimize net consumption of good quality water, it is necessary that effluent waters be treated for reuse within the plant. Six specific conceptual designs of integrated water treatment plants will be

made, and it will be shown how the various water treatment plants can be operated, on a demonstration plant size, so as to gather information for improved designs when commercial-size plants are built. Whereas designs will be provided using the best-known standard technology, additional designs will also be made using novel and innovative technologies. If the return can be shown to be adequate, specific research in these technologies will be recommended.

**RECENT WORK AND ACCOMPLISHMENTS** — Progress has been made on specific treatment designs, particularly biochemical treatments, boiler-feed water treatments, phenol purification, and cooling water control.

**PLANS FOR THE COMING YEAR** — The contract will be completed in 1978. All other specific treatment designs and six integrated plant designs will be made.

## ***LOW-BTU GASIFICATION***

### **SYNTHESIS GAS DEMONSTRATION PLANT PROGRAM**

W.R. GRACE & CO.  
DOE - \$164,278,000; W.R. Grace - \$154,080,000  
9/77 - 9/83

**OBJECTIVES** — This program is designed primarily to promote the use of high-sulfur agglomerating coal as a substitute for oil and natural gas in industrial applications so as to encourage the widespread commercial utilization of low-Btu gasification by the mid-1980's. Specifically, the project undertaken jointly by DOE and Grace encompasses the conceptual design of both commercial and demonstration sized plants to convert coal to synthesis gas for the production of ammonia and, if economically feasible, the subsequent final design and operation of the demonstration sized plant. The program includes process selection, design, and evaluation of the plant to demonstrate the feasibility of converting coal to synthesis gas. If proven feasible, the program will also include demonstration plant construction, testing, and operation. The program is divided into three phases spanning a period of 72 months as follows: Phase I, program development and conceptual design, includes site evaluation, environmental assessment, and the conceptual design of commercial and demonstration sized plants. This phase has a 21-month duration. Phase II, demonstration plant final design, involves procurement and construction of a demonstration plant capable of producing synthesis gas for subsequent conversion to a nominal 1200 t/d of anhydrous ammonia. This phase will have a 37-month duration. Phase III, demonstration plant operation, includes operation of the integrated facility (Syngas plant and ammonia loop) and the collection, evaluation, and reporting of data. Phase duration is 27 months.

The importance of expanding the utilization of coal as a substitute for natural gas and fuel oil lies in the uncertain supply situations. Currently, the U.S. ammonia industry is almost completely dependent on natural gas with each ton requiring approximately 35,000 scf for its production. Again, this year the Federal Power Commission estimates a shortfall of natural gas supplies that may lead to short-term natural gas curtailments to the industrial sector. While Grace and many other ammonia producers are taking steps to offset such curtailments, it is realized that benefits from such measures are more than likely only temporary in nature. Conditions suggest that the ammonia industry will experience a critical natural gas shortage in the next decade if it is to sustain its

production growth in the face of dwindling natural gas supplies. Coal may be the most likely feedstock, for new reliability ammonia capacity. Although some foreign countries use coal as feedstock for ammonia manufacture, most use first generation processes which appear unrealistic for the U.S. due to restrictions on coal type and low gasifier operating pressure ranges. Some general advantages of higher pressure are lower overall power requirements, greater coal gasification capacity per unit volume, efficient heat recovery from downstream processing, and smaller, less expensive (up to a point). In selecting coal gasification technology for the demonstration program, the following are major evaluation criteria: adaptability of the process to high-pressure operation, mechanical reliability of the coal and oxygen feed and the ash discharge systems, ability of the process to gasify agglomerating coals, and overall high thermal and energy efficiency of the integrated design.

**RECENT WORK AND ACCOMPLISHMENTS** – The bulk of technical work will be completed during the remaining months of Phase I. A trade-off study plan for the gasifier operating pressure study is being developed. Implementation of this plan will enable the assessment of the most favorable gasifier operating pressure from both mechanical and economic viewpoints. Work is also proceeding in the development of heat recovery parameters, initial coal preparation specifications, and preparation of major equipment lists.

**PLANS FOR THE COMING YEAR** – Work will be undertaken on process design, specifically in the development of process parameters. Site confirmation activities will begin, including data collection for the purposes of developing an environmental impact report. Also to be performed is an economic evaluation as to the feasibility of the proposed demonstration plant.

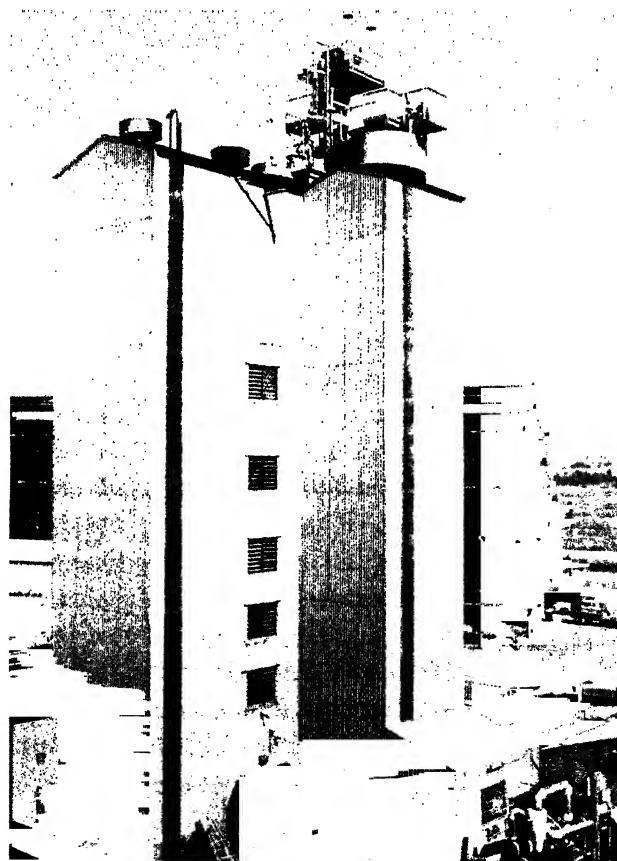
#### **INDUSTRIAL LOW-BTU FUEL GAS DEMO PLANT PROGRAM**

**MEMPHIS LIGHT GAS AND WATER**  
DOE - \$92,966,316; Memphis Light - \$86,500,000  
9/19/77 - 5/79 (Phase I only)

**OBJECTIVES** – This Industrial Low-Btu Fuel Gas Demonstration Plant Program will demonstrate the technical feasibility and economic viability of converting coal, in an environmentally acceptable manner, into a low-Btu gas with a heating value of about 300 Btu/scf, using the U-Gas process. The product will be distributed within a 12-mile radius in the Memphis, Tennessee, area to industrial customers who face natural gas supply curtailment. Specific objectives of the program include conducting process analysis, design, construction, testing, operation, and evaluation of this plant. About 2800 t/d of coal will be converted to 175 million ft<sup>3</sup> gas, equivalent to 50 billion Btu's. The U-Gas process was developed by the Institute of Gas Technology (IGT), Chicago, Illinois. The

**RECENT WORK AND ACCOMPLISHMENTS** – Various aspects of the U-Gas process have been demonstrated by the IGT on a pilot plant scale. Work on the ash agglomeration concept with a coal feed has been conducted at the pilot plant over the past year. A technical support program, including operation of this existing U-Gas pilot plant on the candidate coal for the demonstration plant, is included in the demonstration plant contract to provide process data and to confirm the design basis for the plant. In September 1977, DOE contracted with Memphis Light Gas and Water (MLGW) to conduct Phase I of the demonstration plant program. This phase incorporates the conceptual design and evaluation of a commercial plant, as well as process and mechanical design of a demonstration plant. In addition, this phase of work includes the evaluation and selection of a suitable site for the plant, an environmental analysis, and planning for Phases II and III of the overall program, which include construction and operation of the demonstration plant.

**PLANS FOR THE COMING YEAR** – Phase I, development and conceptual design, is scheduled to last for 20 months. While the current contract is only for Phase I of the program, the entire project is scheduled to last for 72 months, the last 52 of which will be taken up by Phase II, demonstration plant final design, procurement and construction, and Phase III, demonstration plant operation. MLGW will also subcontract with Foster Wheeler Energy Corporation, as architect and engineer, and Delta Refining Company, to provide technical assistance and to operate the demonstration plant. Efforts during the next 12 months of Phase I will include continued design activities, site evaluation, environmental analysis, procurement planning, planning for Phases II and III, and operation of the U-Gas pilot plant in support of the design phase.



*U-Gas Process Developed at Pilot Plant Scale in This Facility*

## COMBINED-CYCLE TEST FACILITY (POWERTON)

COMMONWEALTH RESEARCH CORPORATION

DOE - \$173,500,000; GROUP\* - \$51,000,000

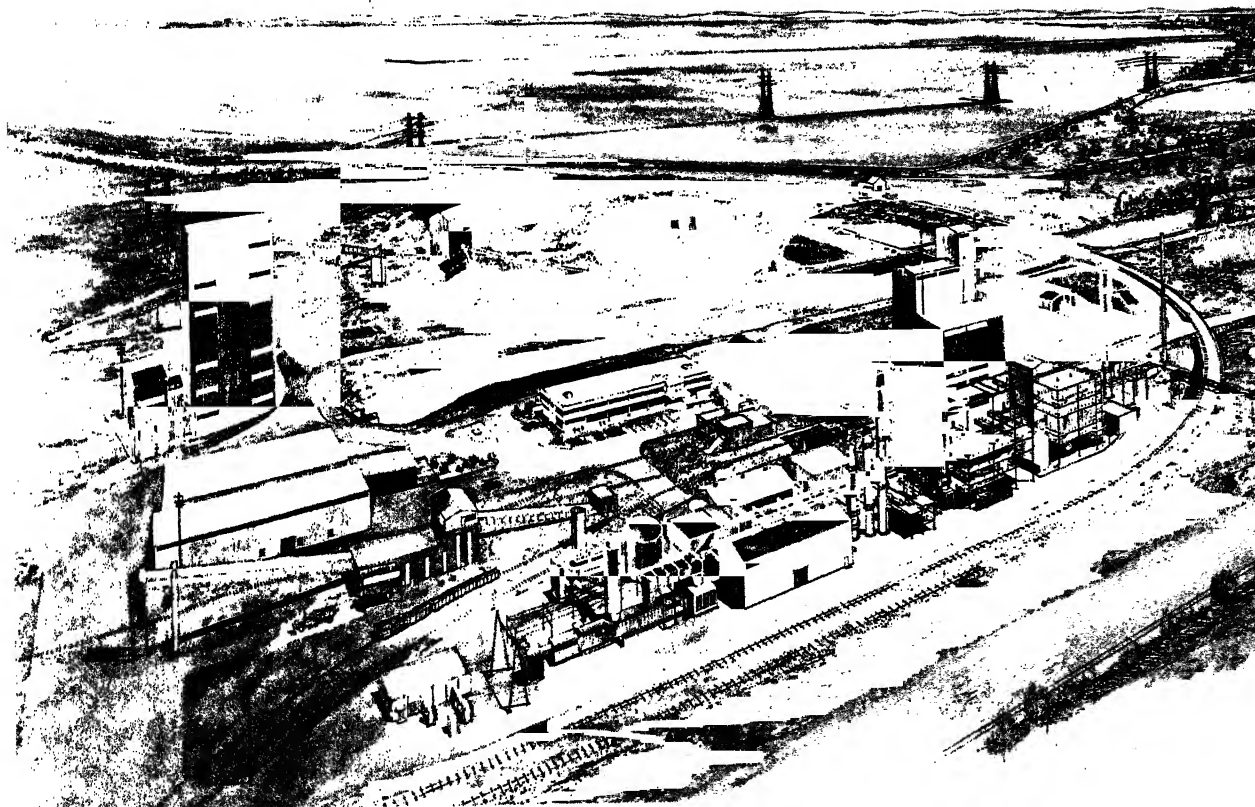
16/5/77 - 9/30/78

**OBJECTIVES** – This activity involves the design, construction, and operation of a coal gasification combined-cycle test facility (Powerton) to produce a low-Btu gas for fueling a gas-turbine generator system that exhausts to a heat-recovery steam generator. Some of the key features of this facility are the provision of space on site for the testing of advanced systems; the in-place coal-handling facilities (480 t/d); the equipment for handling waste and effluents; and the ability to accept full-scale plant output from a gasification system. The 1700-Mw power plant on site permits the use of the produced gas as fuel, or its conversion to electricity and actual use. This arrangement is designed to reduce both the cost and time of bringing new ideas to commercialization. A major goal is to determine the applicability of this system to future large power stations. Gas cleanup methods also will be investigated. In addition, this plant is to serve as a unique and flexible test site for evaluating new coal gasification systems, advanced turbines, fuel cells, and other conversion devices.

**RECENT WORK AND ACCOMPLISHMENTS** – This program has its basis in earlier economic evaluations (beginning in 1972) of various coal gasification and energy conversion systems for the production of electricity. Full-scale detailed engineering was started in May 1977. Construction of the Powerton Plant at Pekin, Illinois, is planned to begin in the first quarter of FY 1979. About a third of the project engineering has been completed. Fluor Engineers and Constructors, Inc. is performing the engineering, procurement, and construction. Lurgi Kohle and Mineralotechnik are providing the process design package for gasification and gas cleanup, and American Lurgi is providing detailed engineering.

According to present plans, the gasification facilities will consist of two 4-meter-diameter Lurgi gasifiers with one gas quench and cooling train common to both. Each gasifier will be a counterflow moving bed reactor to which coal will be fed to the top of the vessel and hot ash removed from the bottom. The counterflow design promotes natural zones for the basic gasification reactions, and allows utilization of the sensible heat of the gas for coal drying and preheating to achieve a high gasification efficiency. The crude gas containing dust, tar, oil, naphtha, excess steam, and various contaminants will be quenched with water in a wash cooler. The saturated and cleaned gas then will pass to gas coolers and the water will be routed to a saturator downstream, thereby transferring heat and water vapor from the crude gas to the clean product gas. This circulating water system will constitute a regenerative form of cooling and heating that conserves the thermal energy in the product gas. The unit will be designed so that hot crude gas will first pass through a prewash scrubber for removal of cyanides and other water soluble contaminants and then through a second scrubber, where desulfurization will be effected. Regeneration of rich solution will be accomplished in a third vessel. The tar separators will be of special design to separate streams of tar, oil, and an

will then flow through the heat recovery steam generator to generate process steam and be vented to the atmosphere. Additional support facilities will include ammonia removal, sulfur recovery by the Stretford process, coal handling, cooling water, plant and instrument air, and flare water treating.



*Powerton Station: Gasification Combined-Cycle Test Facility*

**PLANS FOR THE COMING YEAR** — The design of the facility will be completed. The management of the entire program is being re-examined and will be revised as necessary. In addition, a decision on actual construction of the facility is scheduled late in the fiscal year. The original scope of the program includes a 3-year operation plan, the primary goals of which are to verify that the low-Btu gas produced from six different test coals can be reliably burned; establish the emission levels for each coal; demonstrate the integrated operation of the components; investigate the response characteristics of the plant to electrical system load variations; and obtain design data required for construction of a full-size commercial facility. Specific future tasks include the addition of: second- and third-generation gasifiers, some of which will be oxygen blown, and of high-temperature combustors and turbines.

## LOW-BTU GASIFICATION OF COAL FOR ELECTRICITY GENERATION

COMBUSTION ENGINEERING, INC.  
DOE - \$18,000,000; Combustion Engineering - \$9,000,000

**OBJECTIVES** — This program will develop an atmospheric pressure airblown entrainment gasification system for making a clean low-Btu fuel gas from coal. The gas made commercially by such a system would be suitable for firing utility boilers and gas turbines in an environmentally acceptable manner, and would enable electric utilities to consider adoption of combined steam/gas turbine cycles as a means of plant efficiency improvement in conjunction with the use of coal gasifiers. The immediate objectives of the work are to successfully operate the 5-ton/hour coal gasification process development unit (PDU) located at Windsor, Connecticut, and to complete the test program planned for the PDU.

**RECENT WORK AND ACCOMPLISHMENTS** — Construction of the PDU was completed during FY 1977, and a formal dedication of the facility, attended by state and local dignitaries and officials of DOE, the Electric Power Research Institute (EPRI), and Combustion Engineering, was held on October 18, 1977. Shakedown operation of various components and subsystems of the PDU progressed steadily during the last stages of construction and continued into the first quarter of FY 1978. Experimental work on eleven of the twelve R&D tasks comprising Phase I of this program was completed during FY 1977, and final reports on four of these tasks have been published by DOE. Final reports on all but one of the remaining R&D tasks were in final stages of publication at the end of the year. The R&D tasks for this program covered a variety of subjects including metallurgical and refractory considerations for the process, dynamic flow modeling and mathematical modeling, hazard analysis, environmental considerations, commercial plant studies and PDU operation/test program planning. The last R&D report, Commercial Plant Studies, will be issued early in 1978.

**PLANS FOR THE COMING YEAR** — Initial attempts to fire coal in the PDU gasifier are planned for December 1977. Initial gas-making operation of the gasifier, which involves operation of the entire PDU as a complete system, is expected during January 1978. The planned test program for the design Pittsburgh Seam coal consists of two phases: a parametric test program in which one important variable at a time will be changed, and a sustained operation trial of the plant in which an attempt will be made to keep the entire PDU on stream for a period of 3 months continuously to demonstrate operability and reliability of the plant equipment.

## ADVANCED COAL GASIFICATION SYSTEM FOR ELECTRIC POWER GENERATION

WESTINGHOUSE ELECTRIC CORPORATION, GENERATION SYSTEMS DIVISION  
DOE - \$34,966,000; Industry Team - \$4,053,183  
8/9/72 - 8/8/81

**OBJECTIVES** — A coal gasification system is to be developed that can supply a low-Btu fuel gas to operate modern utility gas turbines and combined cycle generating systems while processing a variety of domestic coals, including caking sulfur-laden varieties, in compliance with all environmental standards. The gasification system must meet a highly diverse set of specifications: it must be capable of accepting a wide variety of run-of-mine coals of variable size and composition; it must provide a gas which has properties within the tolerance band of a modern gas turbine; and it must



be efficient, reliable, and economical when compared with alternate methods of converting coal to electric power. The gasification system may also be operated with oxygen instead of air to produce medium-Btu gas.

**RECENT WORK AND ACCOMPLISHMENTS** — Since the beginning of 1976, work has been directed to development of the gasification system through operation of the 15-ton/d PDU and through supportive bench scale laboratory testing and analysis. The advanced fluidized-bed gasification process consists of two main reactors: a devolatilizer and a gasifier. The devolatilizer accepts a fresh coal feed and decakes and devolatilizes it at nominally 1600°F in a recirculating fluidized bed to produce a free-flowing char, essentially free of volatiles, that can be fed to the gasifier. The gasifier combusts the char in an air jet; steam is introduced to gasify the carbon and to moderate the temperature to nominally 2000°F. At this temperature and under the recirculating conditions in the fluidized bed, ash particles agglomerate and are removed from the defluidized zone at the bottom of the bed. Each reactor system was to be operated independently prior to its operation in an integrated system. The feasibility of the devolatilizer was amply demonstrated in 1976 through sustained operation of the unit with high caking coals. In 1977 the primary emphasis was directed to evaluation of the gasifier reactor. Of particular concern was the design and operation of the combustor and the ash agglomerator. A series of sixteen highly successful test runs exceeding 1100 hours of operation at temperature and pressure and with continuous solids feed and withdrawal were conducted using a variety of feedstock materials. These materials included coke breeze from Pittsburgh seam coals; char previously formed in the PDU devolatilizer from Indiana No. 7, Pittsburgh, and Upper Freeport seam coals; and char produced by the FMC COED process from both Utah and western Kentucky coals. In addition, a number of coals were processed directly in the unit without pretreatment by devolatilization or oxidation. These include Wyoming sub-bituminous coal, Indiana No. 7 coal, and Pittsburgh seam coal. During these tests the reactor was operated at 1750° to 2100°F and 225 psig, at a freeboard velocity of 2.0 to 3.2 ft/sec, and at feed rates from 500 to 1100 lb/hour. Ash agglomerates were produced from each char feedstock and were continuously withdrawn at levels of 45 to 97 percent ash. Separation of ash and char in the bed were distinct and controllable. Start-up by autogenous char combustion with air; continuous combustion, gasification, and agglomeration; and reliable and safe shut-down were achieved for all feedstocks. These tests demonstrated that the gasifier concept was feasible and can be operated efficiently and reliably. Specifically, the accomplishments include: (1) process design was demonstrated with various coals and chars having widely varying reactivities and ash characteristics, (2) carbon utilization in excess of 90 percent was repeatedly achieved, (3) heat and material balances and other data for scale-up were obtained, (4) component designs for hardware, including controls, were developed and tested, (5) continuous, reliable, safe, and controlled operating techniques were defined, (6) coal feedstocks were successfully processed in the reactor without pretreatment by oxidation or devolatilization.

The bench scale and analytical support program at the Westinghouse R&D Center provided essential data which helped to make the PDU gasifier tests successful. This work included the evaluation of gasifier geometries in the cold flow model, the development of char reactivity kinetic data using a hot fluidized-bed reactor and Thermogravimetric analyzer, the investigation of ash agglomeration mechanisms in a small agglomerator reactor, and the continued development of models and other analytical tools to define the process.

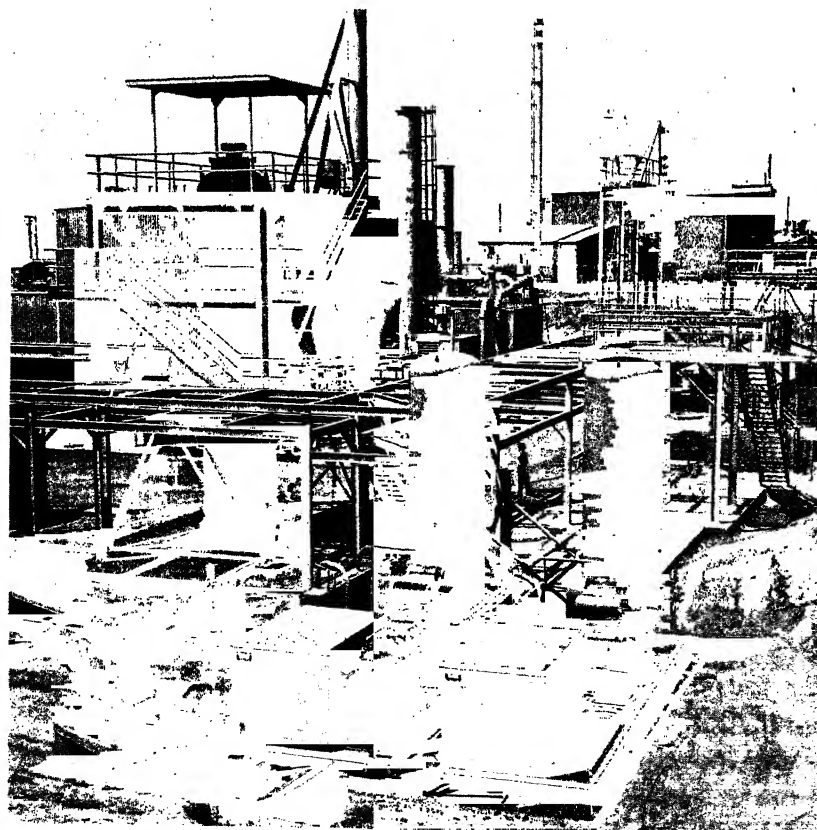


**PLANS FOR THE COMING YEAR** – During 1978 the devolatilizer and gasifier reactors will be integrated and run as a complete system with the gasifier supplying heat and fluidizing gas for the devolatilizer and with the devolatilizer providing char feed for the gasifier. Various control strategies will be tested with a view to typical combined cycle load following applications, and a number of coals will be evaluated as to operating characteristics and product gas composition. In addition, operation of the gasifier with oxygen instead of air will be evaluated.

### **MOLTEN SALT COAL GASIFICATION PROCESS DEVELOPMENT UNIT**

**ROCKWELL INTERNATIONAL CORPORATION**  
**ATOMICS INTERNATIONAL DIVISION**  
DOE - \$9,729,990  
3/28/76 - 12/28/79

**OBJECTIVES** – This program will demonstrate the feasibility of the molten salt coal gasification (MSCG) process for use in the environmentally acceptable generation of electric power. Specific objectives are to design, construct, and operate a Process Development Unit (PDU) which will convert 1 ton/hour of high-sulfur coal into a nonpolluting low-Btu fuel gas. The PDU will be operated to demonstrate integrated system performance and provide engineering and cost data for designing full-scale plants. The MSCG process is of interest because it can handle caking coals without pretreatment, does not require fine grinding or restrictive sizing of the coal feed, and produces a gas which is essentially free of sulfur compounds, ash, coal tar, and nitrogen oxides. In addition, a major portion of the heating value of the coal is retained in the product gas.



*Molten Salt Coal Gasification PEDU under Construction*

**RECENT WORK AND ACCOMPLISHMENTS** – Activities during the year were principally concerned with detailed design of the PDU, procurement of components, and construction of the facility. In order to expedite the program, these activities were conducted in parallel. Three construction subcontracts were awarded, i.e., civil, structural, and balance-of-plant. The initial civil and structural subcontracts were completed, and work is currently underway on the larger balance-of-plant construction subcontract. Engineering activities consisted primarily of the preparation of drawings and specifications for the construction subcontracts, the preparation of detailed specifications for items to be purchased or fabricated, and the review and incorporation of vendor data into the plant design. The major engineering accomplishment was the release of the balance-of-plant construction bid package in June 1977. This package included a comprehensive construction specification and almost 200 drawings. The contract for this construction work was awarded September 19, 1977. All major process equipment items were on order by the end of the report period, and approximately one-half had been delivered to the site. The gasifier vessel was delivered and erected in July in conjunction with the structural construction subcontract. Other key items received during the report period included the high-purity alumina bricks for lining the gasifier, the main air compressor system, and 14 process pumps.

**PLANS FOR THE COMING YEAR** – Construction will be completed early in 1978, and the PDU will be started up. It is anticipated that startup activities will be completed by August 1978, and test operations will be underway by the end of the next report period.

#### DEVELOPMENT OF A FAST FLUIDIZED-BED GASIFIER

HYDROCARBON RESEARCH, INC.

DOE - \$4,019,043

7/1/76 - 4/15/79

**OBJECTIVES** – The overall program plan is to develop a low- or intermediate-Btu coal gasifier which uses the principles of fast fluid bed operation to achieve a tenfold increase in throughput and better turndown capability than conventional fluid bed gasifiers. The first part of this study was designed to analyze existing data, determine the operability of existing equipment at Hydrocarbon Research, Inc. (HRI), and develop an optimum process design. Current objectives are equipment procurement and installation, cold flow model tests at City University of New York (CUNY), and construction and operation of the fast fluid bed gasifier itself.

**RECENT WORK AND ACCOMPLISHMENTS** – A process design has been developed and finalized. Existing equipment including the main air compressor has been tested and overhauled where necessary. HRI was successful in obtaining fixed price competitive bids on all major pieces of new equipment including the gasifier vessels, cyclones, pressure lock hoppers, hot valves, and the support structure. The awards were approved and fabrication has begun. As of October 1, 1977, the foundation was poured and cured and arrival of the structural steel was imminent. Detailed engineering on the piping was well advanced. Underground utility piping has been installed and some of the aboveground piping is in. CUNY tests in a cold flow model provided information which helped determine the gasifier design. These tests are continuing and the data they generate will be important in developing optimum operating conditions for the fast fluid bed gasifier.

**PLANS FOR THE COMING YEAR** – Most of this period will be taken up in completing the detailed design, specifying and procuring the remaining short lead time equipment, and completing

construction of the gasifier. Based on our present schedule, fast fluid bed cold flow critical parts testing should begin in April 1978. This will also serve as operator training. We anticipate that the fast fluid bed gasifier will be ready for start-up operation on coal in September 1978.

### GAS GENERATOR RESEARCH AND DEVELOPMENT: TRIGAS PROCESS

BITUMINOUS COAL RESEARCH, INC.

DOE - \$2,687,483

8/21/73 - 10/21/77

**OBJECTIVES** – A multiple fluidized bed gasification process for the production of low-Btu fuel gas will be developed. The process is designed to gasify a broad range of coals and to avoid the need to remove tars and liquids from the product feed gas. The current work focuses on the operation of a Process and Equipment Development Unit (PEDU) designed for a coal-feed rate of 100 lb/hour. Data obtained on this experimental PEDU will provide information required to scale-up to a commercial size process.



*Tri-Gas PEDU Control Panel*

**RECENT WORK AND ACCOMPLISHMENTS** – The PEDU construction was completed in late 1975. The experimental unit is designed for operation at pressures up to 350 psig and consists of three separate fluidized-bed reactors. Stage 1 serves as a decaking/devolatilization reactor. Stage 2 is the gasifier and is fed with char and off-gas from Stage 1. Stage 3 consumes residual char from

Stage 2 and provides heat and fluidizing gas for Stage 1. Mechanical checkout and shakedown of the equipment was completed. Testing in the Stage 1 reactor was begun using a western noncaking coal (Montana Rosebud). Correlations were developed for the properties of the product char as a function of Stage 1 operating temperature. Stage 1 tests were also conducted using an Illinois No. 6 coal. The first series of tests at low temperature (less than 800°F and in the presence of oxygen) demonstrated the ability to produce a noncaking product char in Stage 1. Further testing at 900° to 1000°F demonstrated the ability to devolatilize the coal while avoiding caking in Stage 1. The product char from this testing was free flowing and did not cake when subjected to laboratory testing at Stage 2 operating temperatures. Testing in the Stage 3 reactor has shown that char made from Montana Rosebud coal can be burned by a mixture of air and steam at 1800°F.

**PLANS FOR THE COMING YEAR** – R&D on the TRIGAS process will continue under a new contract, No. ET-78-C-2798. The PEDU facility will be modified to reduce heat losses through the reactor walls and to provide adequate coal preparation facilities for continuous operation. Testing will continue in Stage 1 and Stage 3 to establish a range of operating conditions for those reactors. Toward the latter part of the program, integrated operation of the three-stage system will be initiated.

### USE OF THE ASH AGGLOMERATING GASIFIER FOR LOW-BTU GAS

INSTITUTE OF GAS TECHNOLOGY  
DOE - \$2,484,318  
7/1/76 - 10/30/78

**OBJECTIVES** – A need exists for an efficient, easily operable process for gasifying coal to produce a low-Btu fuel gas. Such a process could be utilized to provide fuel gas for industrial use. The ash agglomerating process has previously been studied by the Institute of Gas Technology (IGT) for char gasification under a DOE supported program. The process utilizes a fluidized bed and is operated under conditions which favor agglomerating the ash in the lower portion of the fluidized bed. As a result, high utilization of carbon may be achieved while ash low on carbon is discharged in a convenient form for disposal. The existing 4-ft-diameter unit has been studied at atmospheric pressure with coal chars at rates of up to 800 to 1000 lb/hr producing agglomerates with low carbon. The major objectives of this program are to modify the existing gasifier so that raw coal can be dried and stored for long-term operation and to demonstrate the atmospheric pressure gasification of coal under agglomerating conditions. Test runs of up to 30 days are planned. Other objectives include optimizing operating parameters to produce low-ash agglomerates and developing the data necessary as a design basis for a pressurized pilot plant gasifier (nominal diameter of 4 ft).

**RECENT WORK AND ACCOMPLISHMENTS** – During the past year, all modifications and shakedown tests have been completed. Raw subbituminous coal has been successfully gasified without producing condensibles at a 90 percent carbon conversion but without ash agglomeration. The high reactivity of this coal prevented achieving the higher temperatures required for ash agglomeration. Raw bituminous coal has been successfully gasified without producing condensibles or evidence of coal caking in the reactor under very limited operation. Tests are continuing aimed at producing ash agglomerates with this coal. The reactivity is low enough and temperature high enough to allow ash agglomeration, but agglomerates have not yet been obtained to date.

**PLANS FOR THE COMING YEAR** – Illinois bituminous coal tests will continue as necessary to develop the operating conditions to achieve ash agglomeration and high carbon conversion. The design of a pressurized pilot plant gasifier will be completed. The operations phase of the contract will be postponed for at least 6 months while design data are collected in support of another DOE contract, No. EF-77-C-01-2582, with Memphis Light Gas and Water (MLGW). The tests on the MLGW program will continue using a different bituminous coal from Western Kentucky.

#### **GASIFICATION PROJECT - DOUGLAS SITE**

**PIKE COUNTY, KENTUCKY**

**DOE - \$2,894,118; KCER - \$1,500,000; ARC - \$1,750,000**

**4/4/77 - 4/4/82**

**OBJECTIVES** – This program will use coal gasifiers in conjunction with an energy plant, using state-of-the-art equipment, as the sole energy system to support a domestic/commercial development, with provisions to furnish low-Btu gas to industrial users when the need arises. The demonstration facility will also allow collection of technical, environmental, and economic data on a low-Btu gasification system which is used in conjunction with the energy plant to support a development site. All phases of the program, i.e., design, fabrication, installation, and up to 3 years of operation will be evaluated to furnish data and experience as the basis of assessing the utilization of coal gasification in similar applications.

**RECENT WORK AND ACCOMPLISHMENTS** – Extensive process analysis and design has been performed, resulting in the selection of the major items of equipment. Among these are two 6½-ft diameter Wellman-Galusha gas producers with ancillary equipment, two 20,000-lb capacity boilers equipped to burn either low-Btu producer gas, or fuel oil and suitable for later conversion to coal firing, two 570-ton absorption chillers as well as ash removal and coal handling equipment, cooling tower, water treatment equipment, pumps, hot water convertors and various controls and items of instrumentation. Utilizing the “fast-track” method of construction, all of the above-mentioned items of equipment are planned to be “pre-purchased” and, in fact, the gas producers, boilers and chillers have been purchased. Several meetings have been held between representatives of Pike County, Mason & Hanger-Silas Mason Co., Inc., and the various governmental agencies involved to discuss and resolve various aspects of the project, including operational safety, environmental protection, and operation. Of importance among these items is the fact that, to meet the requirements of EPA, it will be necessary to add a desulphurization or “cleanup” system to the producer gas before it can be furnished to future industrial users on the site.

**PLANS FOR THE COMING YEAR** – Work will include completion of final design, procurement of the balance of equipment, commencement of construction, and installation of equipment. Final completion, shakedown, and startup are programmed for the third quarter of calendar year 1979.

#### **COAL-DERIVED GAS FOR FOOD PROCESSING PLANT**

**LAND O'LAKES, INC. (LOL)**

**DOE - \$3,239,242; LOL - \$3,239,242**

**Construction plus 3-year operation**

fuel gas made from coal will be evaluated for spray drying dairy-food in industrial boilers. For spray drying foods, the gas will be nearly

perfectly cleaned and then burned and used for direct heat exchange with the food product. Forty-three t/d of bituminous coal will be converted in a two-stage Wellman Incandescent demonstration gasifier. The fuel gas is made mostly in the hot lower stage. The upper stage is heated by the hot gases from the lower stage—half in direct exchange and half in indirect. The product of the upper stage will be mostly a tar at 200° to 300°F, carried off by the gas from the lower stage. The complete project is expected to take 5 years.

**RECENT WORK AND ACCOMPLISHMENTS** — A draft contract with Applied Technology Corporation for the A/E work has been submitted to DOE. Potential coals have been evaluated in the laboratory. A modified lignite is being considered as a feedstock. Gas cleanup by the Koppelman high-temperature fail-safe filter is being developed at Stanford Research Institute (SRI). The efficiency of this filter in the collection of flyash is equal to that of a bag filter in laboratory tests. Based on these data, specifications for such a filter to clean the 1200°F fuel gas and for the gas itself were developed. These may be modified later. Interpoll, Inc., has completed their Environmental Impact Assessment, and it has been forwarded to Mitre Corporation who will prepare an integrated report.

**PLANS FOR THE COMING YEAR** — The small-scale tests of gas clean-up will continue using pilot modules to be designed and tested at SRI and in existing test gasifiers. Tests in the plant spray dryer will be made to show that trace SO<sub>2</sub>, equivalent to the sulfur in gas from coal added to the drying air currently being used, has no effect on the milk-derived products. Major systems for the demonstration gasifier will be designed and major equipment will be ordered. Completion of the gasifier and retrofit of the boilers and dryer are now estimated at 18 months from final approval of contract with the A/E subcontractor, Applied Technology Corporation.

## LOW-BTU COAL GASIFICATION APPLICATION

### UNIVERSITY OF MINNESOTA

DOE - \$3,197,278; University of Minnesota - \$2,680,362

10/5/76 - 5/14/81

**OBJECTIVES** — The coal gasification program at the University of Minnesota, Duluth Campus, Heating Plant is designed to demonstrate the cost and environmental effectiveness of converting gas/oil fired boilers to a coal-derived fuel using a two-stage gasifier. Many institutional, industrial and commercial boiler plants, such as the University of Minnesota Plant, have been designed for firing only gas and oil and cannot economically be converted to coal firing. The two-stage gasifier, when operating at full capacity, will have an anticipated output of 328,882 scf (60°F) of gas with a higher heating value of 166 Btu/scf (60°F). The coal requirement, on maximum load, will be 3 t/hr. The two gas streams will have the following cleanup equipment: the bottom gas (1200°F) from the coking section will flow to a refractory-lined cyclone for removal of carryover char particles; the top gas (250°F) from the volatilizing section will flow to an electrostatic precipitator for removal of tar oil. After cleanup, the two gases will be combined and piped directly to two water tube boilers. The combined gas will have a temperature of 750°F.

**RECENT WORK AND ACCOMPLISHMENTS** — Design of the 10-ft FWEC-Stoic gasifier and its supporting systems has been completed, and all equipment and construction work has been purchased or is under contract. Construction has started on the plant addition with all below-ground work completed.

**PLANS FOR THE COMING YEAR** – Completion of the installation is projected for late June. A 2-month startup and shakedown period is anticipated with the system going on line late in August 1978. Then, a 3-year program of demonstrating and evaluating the gasifier will be initiated, testing operation of the system on different types of coal.

## OPERATIONAL LOW-BTU COAL GASIFICATION TECHNOLOGY

ACUREX CORPORATION/AEROTHERM DIVISION  
DOE - \$595,000; Acurex - \$88,000; Glen-Gery - \$596,700  
11/76 - 10/78

**OBJECTIVES** – This program will accomplish the following objectives: demonstrate the applicability of low-Btu gas derived from anthracite coal for firing a continuous tunnel kiln used for making bricks, demonstrate the cleanliness of low-Btu gas derived from anthracite coal—the gas produced from anthracite has very few tars and oils—which can be used directly without elaborate and expensive cleanup devices, demonstrate the operational efficiencies of a fixed bed atmospheric gasifier (Wellman-Galusha unit) operation on anthracite under various load conditions, evaluate the economics of operating a Wellman-Galusha gasifier on anthracite coal, demonstrate the safety requirements for handling low-Btu gas derived from anthracite coal, demonstrate the viability of anthracite coal gasification for industrial usage throughout the northeastern region of the United States, provide detailed process data to Glen-Gery Corporation to enable optimization of their operation and fuel costs, and acquire data that directly relate the operation of a Wellman-Galusha gasifier to the local environment.

**RECENT WORK AND ACCOMPLISHMENTS** – Installation of the second Wellman-Galusha gasifier at the York, Pennsylvania, plant of Glen-Gery Corporation is complete. Installation began During January of 1977. The gasifier will come on line in mid-October 1977. Three hours after the bed was lit the gasifier was providing fuel to the tunnel kiln; the maximum gasifier capacity is ~ 25MM Btu/hour. The producer gas Btu content is about 140 Btu/scf. Installation of the gasifier monitoring system is 90 percent complete. Spot checks of the gasifier's operation and gas quality show steady operation. In this application, gas cleanup other than the standard cyclone on the Wellman-Galusha gasifier is not required. A gas pump has been installed to better control the gas line pressure with the two gasifier units manifolded together supplying two kilns.

**PLANS FOR THE COMING YEAR** – The program will focus on the acquisition of detailed operational data. In addition operational perturbations will be made to study the gasifier's performance under various non-standard operational conditions. Variations in coal size will be one of the major operational parameters studied. Presently, the gasifier operates on a 50/50 mixture of pea and buckwheat sized anthracite. Variations in coal size will be made to the smaller sizes such as rice. Air saturation temperature will also be varied to study the effect of this parameter on the bed operation and producer gas quality. Load factor variations will also be studied as they relate to the gasifier's efficiency and gas quality. The economic study to be performed will use all utilities, labor, and fuel costs including labor and hardware costs associated with the yearly maintenance which occurs during the one week of down time in the summer months.



## LOW-BTU COAL GASIFICATION SYSTEM

### GENERAL REFRACTORIES COMPANY

DOE - \$2,122,500; GRC - \$2,122,500

9/27/77 - 9/26/79

**OBJECTIVES** — Fuel gas made from coal will be evaluated for use in the firing of refractory kilns. Approximately 2 t/hr of coal will be fed to a two stage Woodall-Duckham gasifier. After separation of tars and particulate matter the gas (210 Btu/ft<sup>3</sup>) will be distributed to heat existing refractory kilns. The contract is for a 36-month period to design, construct, retrofit, checkout and operate to collect data with which to evaluate the process.

**RECENT WORK AND ACCOMPLISHMENTS** — In FY 1977, a cooperative agreement has been negotiated and is undergoing final processing before signatures.

**PLANS FOR THE COMING YEAR** — In FY 1978, the cooperative agreement will be signed, major subcontract signed, and preliminary engineering accomplished.

## LOW-BTU GAS/FIXED-BED GASIFIER PROGRAM

### MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$3,566,000

7/1/75 - Continuing

**OBJECTIVES** — The Morgantown Energy Research Center (MERC) 42-inch stirred, fixed-bed gasifier project will provide a reliable and steady state source of clean low-Btu gas for use in evaluating end-use applications of the fuel gas. The gasifier also serves in developing gas cleanup (cold and hot) and waste effluent treatment systems, studying various aspects of low-Btu gas combustion, and testing and evaluating valves and coal feed systems for coal conversion plants. The use of gasification products for clean-air power and industrial heating requires gaseous cleanup. The combination of test programs will provide information needed to develop commercial plants.

**RECENT WORK AND ACCOMPLISHMENTS** — In four operating periods, mechanical and instrumentation improvements as well as operational features of a prototype cleanup system, operating on a sidestream of producer gas, were tested. Winterization and reliable mechanical operation of the gasifier have been demonstrated. Design verification tests on the sidestream gas conditioning unit have shown: (1) condensing out high-boiling tars in the dry state at 300°F removes a major portion of the principal contaminants and uses the least amount of water, (2) pressure decantation of scrubbing liquor coupled with closed-loop heat exchange is a viable unit operation that both minimizes water-oil emulsions and accounts for the heat in the process gap, (3) COS and CS<sub>2</sub> in producer gas should be hydrolyzed over catalyst to H<sub>2</sub>S before desulfurization since producer gas on combustion will violate constraints for SO<sub>2</sub> emission, (4) an electrostatic precipitator for operation at system pressure and 300° to 350° is needed to protect the COS converter and to quantitatively remove solid particles, and (5) recycled water containing 1-6 percent by weight emulsified light oils is more efficacious in removing alkali metals from producer gas than is once-through water. Using data from these tests, designs have been finalized for a full-flow wet cleanup system. Procurement and construction activities are underway for the system which will be installed and operated in 1978. Water and effluent characterization and treatment studies began to develop design information to achieve environmentally acceptable effluents. Gaseous, vapor, and particulate sampling and



characterization testing is being incorporated into each operating period. Water treatment studies are examining biological oxidation, stripping and carbon adsorption, and a novel wet-oxidation process.

**PLANS FOR THE COMING YEAR** – Construction and installation for a full-flow gas cleanup system will proceed during the coming year. The total system will include water scrubbing, precipitation of particulates, hydrogen sulfide removal in a Holmes-Stretford unit, and final water wash. The operation of the unit will focus on check out of the new systems along with continued characterization of the operating parameters of the gasifier.

### SLAGGING FIXED-BED GASIFICATION

GRAND FORKS ENERGY RESEARCH CENTER

DOE - \$1,540,000

10/1/76 - Continuing

**OBJECTIVES** – This project proposes to establish and demonstrate continuous 5- to 7-day operation of an existing slagging fixed-bed pressure gasifier (of about 1-ton/hour capacity) on a range of coals, including both low-rank western and agglomerating eastern coals. Part of the project includes study and development of controlled and improved operating techniques for use in larger scale units; of factors relating to slagging mode of operation, including refractory life, heat transfer, and slag flow; and of coal specific operating parameters and limitations. Composition will be characterized and the production rate will be established for process by-products and wastes generated in fixed-bed gasification of a variety of coals, including processing and treatment for maximizing water and by-product value recovery. Characterization and control of environmentally related factors associated with plant specific operation, including coal trace element distributions and fate, will be accomplished. A source of coal-specific gasification products will be provided for study to enlarge the data base of the oxygen pressurized fixed-bed gasification process. The principles of operation of the test unit are similar to the commercial dry ash Lurgi process, with the exception of the method of slag removal, which is a modification with important process improvements. The GFERC unit is the only existing operable gasifier of its type in the United States.

**RECENT WORK AND ACCOMPLISHMENTS** – Reactivation of the GFERC test unit, which had been maintained in standby condition for about 10 years, was completed in the final quarter of FY 1976. Initially, objectives based on the short-term tests possible with available GFERC staffing were directed primarily to characterization of generated effluents from low-rank non-agglomerating coals. Activities at the beginning of FY 1977 focused on establishing reliable operation and development of sampling and analytical techniques to characterize and study the condensibles produced in the gas and to establish the mass production rate of these materials. Satisfactory slagging conditions were achieved on lignite. Because of the unique problems associated with high-pressure, low-temperature conditions, a variety of sampling procedures were compared for the gas liquors. Also, in most instances, modifications to state-of-the-art analytical techniques and procedures were required because of the process-specific effluent composition. Ultimately, refinements and improvements were achieved. Operation was conducted primarily on lignite from the Indian Head mine, near Beulah, North Dakota. A significant body of data has been accumulated relating to gas liquor composition and operational parameters. An expanded program plan was approved and funded in the third quarter of the fiscal year. The plan provides for an addition to the GFERC pilot plant facilities for gasification-related auxiliaries, operation and analytical functions provided by

contractor, and construction of a support facility for contractor personnel. Design of facility additions was completed by a local AE firm and contracts awarded for construction. A coal conveying and charging system from storage to gasifier lock hoppers is being designed. A contract with Stearns-Roger, Inc., was activated on May 9, 1977, and design work on gasifier modifications immediately initiated. Modifications to the gasifier include replacing the present batch hopper with a double lock hopper system to stabilize operation; installing a stirrer in the top section of the gasifier shaft to break agglomerates formed in tests on agglomerating coals, and upgrading auxiliaries and data collecting capabilities. Contractor personnel will operate the pilot plant and perform routine analytical functions.

**PLANS FOR THE COMING YEAR** – Operation with present configuration will be directed to achieve the following objectives: (1) establish slagging operability on selected non-agglomerating western coals; (2) continue to develop, refine, and verify sample collection and analytical procedures for gas, liquids, and solids; (3) study, test, and compare hearth plate refractories and slag flow considerations; and (4) compile data base on effluent characteristics, mass rates, and balances on tested low-rank coals. Operation of the gasifier will be suspended in June 1978 to permit construction activities relating to coal handling and gasifier modifications. It is expected that operational capability will be restored in November 1978.

#### SLAGGING FIXED-BED GASIFICATION

STEARNS-ROGER, INC.  
DOE - \$4,517,000  
5/9/77 - 5/9/80

**OBJECTIVES** – This project is designed to provide engineering design services, procure and manage installation of modifications, to operate the pilot plant slagging fixed-bed gasifier at GFERC, and to provide routine analytical services required to evaluate operating parameters and characterize effluents produced in gasification of a variety of both low-rank and agglomerating coals. The goal is to achieve the capability to operate continuously for periods of 5 to 7 days.

**RECENT WORK AND ACCOMPLISHMENTS** – Design work under this contract was initiated on May 9, 1977, on gasifier modifications. These modifications include (1) replacement of the single coal lock with two lock hoppers to provide stable feed flow; (2) addition of a stirrer to top section of gasifier to break up agglomerates formed during operation on eastern coals; (3) addition of a second slagging section for the gasifier, essentially a duplicate of the existing unit; (4) addition of on-line gas analysis instrumentation; (5) upgrading or replacing gasifier auxiliaries; (6) addition of a data logging system; and (7) addition of an incinerator for disposal of gas liquors generated. Progress of these efforts is on schedule, and this phase of the project is to be essentially completed by November 15, 1978. The project manager, one project engineer, and administrative staff for operation of the gasifier were on-site in third quarter of the fiscal year. Candidate operators from the CO<sub>2</sub>-Acceptor pilot plant, Rapid City, South Dakota (scheduled to terminate operations at the end of the fiscal year), observed gasifier operations and became acquainted with the procedures.

**PLANS FOR THE COMING YEAR** – Delivery and installation of gasifier modifications will occur in the last quarter of the fiscal year. Shakedown operation will commence near the fiscal year's end, with projected operation scheduled for mid-November. Engineering, analytical, and operational staff will begin transfer from Rapid City, South Dakota, to GFERC during the first quarter. It is

projected that personnel for two-shift operation will be on site by the second quarter. During the first quarter, operation and analytical services will be in transition status, during which time the combined GFERC and SRI staff will perform required functions. All operational and routine analytical functions will be performed by SRI personnel by the second quarter of the fiscal year.

### HIGH MASS FLUX COAL GASIFIER

BELL AEROSPACE TEXTRON

DOE - \$1,025,323

1/30/76 - 2/28/78

**OBJECTIVES** – A high mass flux (HMF) entrained flow gasifier reactor will be developed to economically convert coal to low-Btu gas, using rocket engine combustor principles. The project is directed at miniaturizing reactor volume. By utilizing high efficiency mixing techniques, from rocket engine technology, significant gains in mass throughput can be realized. The resultant effect is a smaller reactor (by one or more orders of magnitude), substantial savings in capital and plant operating costs, and advancement in the state-of-the-art. Successful development of the HMF gasifier will provide the basis for a new family of gasifiers with high economic potential.

**RECENT WORK AND ACCOMPLISHMENTS** – The Phase I feasibility demonstration program was successfully completed and the final report issued. Work on Phase II of the program began with the design and fabrication of two reactors for evaluation during extended run duration tests. One reactor incorporates a swirl air injection system and the other uses a reverse flow air injector. Extensive modifications were made to the test facility to accommodate 1-hr duration tests at coal flow rates of nominally  $\frac{1}{2}$  t/hr. Thermodynamic equilibrium analyses have been conducted to establish the effect of air/coal ratio and coal moisture content upon make gas species. Calculations have been made for the reactions of air with lignite, Montana, Rosebud, and Pittsburgh seam coal. Calculations have also been made to establish the effect of adding steam to the above reactions. The test facility has been checked out and the gasification test program initiated. Initial tests are being conducted at nominally 15 atm pressure using pulverized lignite. Nominal coal particle size is 74 percent through 200 mesh sieve, and the average moisture content is approximately 7 percent. An initial 1-hr duration gasification test has been completed. Operation of the gasifier and test facility has been satisfactory for all tests completed to date. Some slag accumulation has been experienced on the "cold" metal surfaces of the air injector; design changes are planned to eliminate this problem.

**PLANS FOR THE COMING YEAR** – Reactor testing will continue and 1-hr duration tests will be conducted with lignite, Montana Rosebud, and Pittsburgh seam coal. Reactor operating characteristics will be evaluated as a function of run duration and, based upon test results, reactor designs, development plans, and application studies will be updated.

### EXTRUSION FEEDING OF COAL FOR FIXED-BED GASIFIER

MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$500,000

7/1/77 - 9/30/79

**OBJECTIVES** – Coal feeding systems to meet requirements of coal conversion processes are under development. One concept being examined and tested is the dry extrusion of coal into gasifier

pressures up to 1000 psig. Two extruder concepts will be tested on MERC gasifiers. A 5½-inch Ingersol-Rand extruder will be installed and tested on the 42-inch fixed-bed gasifier, and a 2½-inch Egan extruder will be adapted for use on the 16-inch atmospheric pressure gasifier. The goal of this testing will be demonstration of the operability of extruders as coal feed devices for coal.

**RECENT WORK AND ACCOMPLISHMENTS** — Basic plans and schedules have been developed for testing the two extruders. A 2½-inch, 220 lb/hour Egan extruder was delivered at the end of the fiscal year. The extruder will be adapted and tested by MERC with and without additives prior to installation on the 16-inch gasifier. A laboratory program to investigate additives is being defined. The structural design to accommodate the 5½-inch Ingersol-Rand extruder on the 42-inch gasifier is underway and nearing completion.

**PLANS FOR THE COMING YEAR** — The Egan extruder will be bench tested and modified for installation and testing on the 16-inch atmospheric pressure gasifier. A parallel laboratory study of additive effects on extrusion properties will be completed and the results used to define the final operation on the gasifier. A 5½-inch Ingersol-Rand Impco extruder will be delivered to MERC for testing on the 42-inch gasifier. This extruder will be tested on coal prior to delivery by the supplier. The design rates for this extruder are 5 tons/hour at 1500 psig, but testing will be at a 1 ton/hour rate at 300 psig. Structural design for installation of the extruder will be completed early in the year followed by testing on the gasifier.

## DEVELOPMENT OF COAL FEEDERS

INGERSOLL-RAND RESEARCH, INC.

DOE - \$2,642,445

7/1/75 - 10/1/80

**OBJECTIVES** — This program is designed to develop pilot plant size coal feeder devices that can be scaled up for feeding large quantities of coal into pressurized commercial-scale reactors. By two contract modifications during this year, the scope of work was expanded to include two new areas of activity. The program now consists of three basic elements: development of a continuous dry coal screw feeder, development of a dry coal piston feeder, and investigation of the screw feeding of coal under fully plasticized conditions. The screw feeder program involved detailed examination of various coal conversion processes being considered for commercial-scale operation to establish specific feed system requirements for each process. New concepts for coal feeders were to be conceived and evaluated. In addition, two sizes of coal screw feeders will be designed, fabricated and tested to determine system capability and performance as a function of various design and operating parameters. The IMPCO 1500, 5½ in. diameter screw feeder will be installed and operated in the Morgantown, West Virginia, gas producer pilot plant. Feed rates are targeted to be in the range of 0.5 to 5 t/hr. Based on performance of this screw feeder installation, recommendations will be made for the design, fabrication, and testing of coal screw feeder equipment compatible with projected demonstration plants. Independent assessments of the feasibility of three new dry coal piston feeder concepts indicated sufficient promise to warrant a parallel development effort. A three-phase contract amendment was received to explore these concepts. A pilot plant scale feeder of the selected concept will be designed, fabricated, and laboratory tested. The feeder will then be installed and operated in a selected pilot plant. In addition, recommendations will be made for the design, fabrication, and testing of coal piston feeder equipment compatible with projected demonstration plants. The feasibility of delivering fully plasticized coal in spray form and in an acceptable

particle size range against atmospheric and elevated back pressures with the Negri Bossi V-12 screw feeder will be established. The economic merit of coal feed by this method will be assessed and compared to other feed methods. The present program will provide solutions to problems presented by lockhoppers and slurry feed systems, in current use, through the development of equipment capable of the continuous delivery of dry, pulverized coal to the high pressure environment of current gasification processes.

**RECENT WORK AND ACCOMPLISHMENTS** – Various types and sizes of coal were successfully pumped on a continuous basis to elevated gas back pressures with the Negri-Bossi V-12 screw feeder. It was found entirely feasible to seal against such back pressure by taking advantage of the plasticizing properties of the coal or by compaction of the coal particles. The results of this work have guided the design and testing activities for the 5½ in. diameter IMPCO screw feeder. Shake-down testing of the 5½ in. diameter screw feeder as well as the test system were initiated in March 1977. Desired changes made after testing included (1) modifications to provide for a continuously variable speed capability and (2) modifications to permit operation in the injection mode. Testing of this feeder against atmospheric back pressure and up to 300 psig back pressure has now been completed in the extrusion mode (with external heating). Steady state operation at 300 psig back pressure for a period of approximately three (3) hours has been successfully demonstrated. During this test, a feed rate of 300 lbs/hr on Pittsburgh No. 8 coal, was obtained. Activities on the dry coal piston feeder concepts were initiated on October 1, 1977. Preliminary analysis has been completed, areas and components requiring detailed investigation have been identified, and bench-scale models for their evaluation have been designed and fabricated. Design modifications were completed on the Negri Bossi V-12 screw feeder to achieve the higher heat levels required and capture the extrudate spray. The screw and die configurations were selected and procured along with the necessary instrumentation. Shakedown testing of the completed system is now in progress.

**PLANS FOR THE COMING YEAR** – Testing of the 5½ in. diameter IMPCO 1500 screw feeder will continue in the extrusion mode (with external heating). Modifications to the feeder will be made to permit testing under the injection mode without external heating. Emphasis will be placed on devising and implementing techniques for increasing coal output and reducing specific power consumption. A preferred operating mode for pilot plant use will be identified, and a continuous 10-day reliability demonstration will be conducted at a simulated back pressure of 300 psig. Upon successful conclusion of this demonstration an investigation of performance above 300 psig back pressure will be performed. The dry coal screw feeder, with all necessary interfacing and supporting auxiliary equipment, will be shipped to the Morgantown, West Virginia Energy Research Center for mating with the pilot plant gas producer. With respect to the piston feeder concepts, bench-scale screening tests will be performed on critical feeder element concepts to determine feasibility. A preferred piston feeder configuration will be identified from these tests and concurrent analytical studies of geometry optimization, performance, and system dynamics. A pilot plant scale feeder, based on the preferred concept, and delivering up to 48 t/d, will be designed and fabricated including all auxiliary actuation and control systems necessary for the operation of the piston feeder. Laboratory tests will be initiated, under simulated gasifier back pressure conditions to demonstrate overall piston feeder conformance with target performance and operating specifications. Shakedown testing of the revised Negri Bossi V-12 screw feeder is now in process. This work will be followed by systematic test series under atmospheric and elevated back pressures conditions on several candidate coal types to investigate the effect of major test variables on machine and spray nozzle behavior. Power requirements and delivery rates will be determined as well as the physical

condition of the delivered coal. An overall assessment of the feasibility of plasticized coal delivery with the Negri Bossi V-12 screw feeder will be made. If favorable, a limited conceptual design of a commercial-scale unit will be made for economic comparison with other feeders for coal gasifiers.

## COAL FEEDER DEVELOPMENT

FOSTER-MILLER ASSOCIATES, INC.

DOE - \$1,897,513

5/21/75 - 6/30/79

**OBJECTIVES** – Dry coal feed systems will be developed for pressurized coal gasification processes. This objective will be accomplished through the following tasks: (1) develop coal feed injector concepts based on a detailed examination of the system requirements imposed by coal gasification processes, (2) conduct laboratory-scale modeling and critical components testing to determine concept feasibility, (3) design, fabricate, and test coal feed injector equipment compatible with existing and projected coal gasification pilot plant requirements, and (4) make recommendations for the design, fabrication, and testing of coal feed injector equipment compatible with projected demonstration plant requirements. A critical problem with all gasification processes which operate above atmospheric pressure is the delivery of coal to the gasifier. The primary means now employed to feed high-pressure gasifiers (>100 psi) are lock hoppers and slurry feed systems, both of which have serious shortcomings rendering them undesirable choices for commercial gasification processes. In most applications, slurry feed systems, although reliable, require large amounts of energy to evaporate the slurrying liquid or extensive equipment to separate the coal from the liquid at high pressure. With lock hoppers, on the other hand, there is a serious question that satisfactory reliability can be achieved in high-pressure applications. Problems with present feed systems can be overcome to some degree in pilot plant applications by essentially ignoring economic considerations. This is acceptable in a pilot plant where the primary objective of the operation is to experiment with process dynamics. However, the development of dry coal feed systems which can overcome the shortcomings of systems now used for feeding pressurized gasifiers is essential if coal gasification is to become a viable commercial activity.

**RECENT WORK AND ACCOMPLISHMENTS** – The primary objective of the program effort in 1977 has been to fabricate and test the three FMA pilot plant feeder prototypes. These include a centrifugal feeder, a fluidized piston feeder, and a linear pocket feeder (LPF). A batch-type test "loop" was designed and built for testing these feeders. Many components of the test loop were government furnished, having originally been fabricated for an OCR feeder development program conducted by Bituminous Coal Research. Testing of the centrifugal feeder began in November 1976. The prototype feeder has achieved the design goal of 200 psig at a feed rate of 1.7 t/hr with 70 percent through 200 mesh coal. Considerable theoretical work has been done in parallel with the experimental program. The theoretical predictions are in good agreement with the experimental results. The fluidized piston feeder has successfully fed 150 lbs/hr of 70 percent through 200 mesh coal at 900 psig. Further development work is required on the feeder valves. Development of the prototype device has been suspended due to funding limitations. The linear pocket feeder has experienced testing delays due to a 4-month slippage in the delivery of the pressure vessel components of the feeder. The device has fed -3/4 inch coal at a rate of 5 tons per hour. Initial testing at pressure without coal has shown, as predicted theoretically, that the leakage rate is significantly reduced as the conveyor speed increases. The present design utilized

state-of-the-art materials for extreme abrasive service conditions. "Foster-Miller's Development of Dry Coal Feed Systems" a paper presented at the DOE sponsored Conference on Coal Feeding Systems held at the California Institute of Technology in June discussed in detail the background, design, and development of FMA feeder systems.

**PLANS FOR THE COMING YEAR** — Program effort during FY 1978 will be devoted to five major areas. (1) Design and fabricate a batch type test loop capable of running for 1 hour at a feed rate of 5 t/hr with coal sizes from -3/4 inch to 70 percent through 200 mesh. The maximum operating pressure of the test loop will be 1000 psig. All ancillary equipment (coal handling equipment, gas handling system, safety devices, etc.) will be included. (2) Design, fabricate, and test a centrifugal feeder prototype to feed 5 t/hr of coal against 500 psig. Varying coal size distributions will be experimented with. (3) High-pressure testing will be conducted with the LPF. The goal of the program is to determine gas leakage rates as a function of gas pressure and conveyor chain velocity. (4) Redesign, fabricate, and test a new LPF with emphasis on ease of maintenance, extended wear life, and reliability. (5) Conduct 1000-hour wear tests using the present LPF and no pressure in order to select proper materials for critical areas.

### **COAL FEEDER DEVELOPMENT PROGRAM**

**LOCKHEED MISSILES & SPACE COMPANY, INC.**

**DOE - \$2,591,640**

**6/26/75 - 9/26/79**

**OBJECTIVES** — The reliable feeding of large quantities of dry pulverized coal into pressurized reactors poses a challenging problem. Presently, some installations are using lockhoppers. However, at the higher operating pressures and for large throughputs, which will require large valves, these systems are beyond the state-of-the-art, or at best inefficient. Based on the available evidence, the reliability of these systems will also impact plant operation. Slurry systems using either process-derived oil or water are in use or being contemplated. The slurries must be dried before further processing which has not been demonstrated for large-size applications. This drying step clearly is detrimental to the overall plant efficiency. At present, no system is commercially available to feed large quantities of dry pulverized coal into pressurized reactors at the large rates projected for future gasification plants. The objective of the program is to generate sufficient analytical and test data to enable the confident design and fabrication of coal feeders which are compatible with demonstration plant requirements and commercial applications. The program is being performed in three phases: Phase I, selection of concepts, was designed to review potential candidates and equipment, synthesize designs, assess fundamental problem areas, and define laboratory evaluation techniques. Phase II, laboratory-scale feeder development, involved building and testing of laboratory size feeders in a continuous loop test facility. The data resulting from laboratory testing



kinetic extruder and the fluid dynamic lock were being manufactured, and the equipment required to establish a test loop was being fabricated. Extensive testing, using both gaseous nitrogen and steam as the driving medium, was carried out with the bench-scale ejector unit. The data acquired were used to refine the design procedures and to establish the capability to predict ejector performance and scaleup. A laboratory-scale unit was built and tested to verify this capability. The methods developed have been used to evaluate the potential of ejectors in pilot and demonstration plants. Based on the present status, it appears that ejectors are well suited as booster or topping stages in high-pressure systems and that the use of steam-driven ejectors should be considered if found to be compatible with the coal-conversion process. Initial testing with the first unit built indicated that the kinetic extruder was able to transfer coal across the pressure barrier. These tests also indicated that major changes in hardware design were required before extensive testing could be accomplished. The main problems were caused by coal dust infiltrating the space between the rotating hollow power shaft and the stationary coal feed tube. The redesign effort, resulting in the Kinetic Extruder Model No. 2, has solved all major problems and successful testing was accomplished. The data verify that the design procedures developed and the performance predictions made are accurate for scaleup to pilot plant size and trade studies.

Tests conducted during the first year had shown the basic feasibility of moving a column of steel balls and coal against a pressure gradient of up to 1.6 psi/ft while maintaining a gas seal. During the reporting period, an evaluation was made of a coal-loading and a ball conveyor letdown concept. These components of the ball conveyor system should be tested before an all-up system is built. The ball conveyor is shown to be capable of high feeding rates against low pressures. Tests performed with the fluid dynamic lock have verified that no additional pressure rise can be obtained across a unit because of the high-particle density. The need for narrow disk spacing limits application to very fine coal grinds, and the limited pressure rise per stage forces the use of many stages, increasing power consumption and equipment cost. The device is not recommended as a coal feeder but should be considered as a recompression unit for recirculating fluidizing gases in reactor vessel.

**PLANS FOR THE COMING YEAR** – Candidate feeder concepts with a strong probability of commercial-scale viability are being designed. The concepts under study include a single-stage kinetic extruder, a two-stage kinetic extruder, and a hybrid system consisting of a first-stage kinetic extruder and a second or topping ejector stage. Elements of these systems will be fabricated and evaluated in the existing test loop.

## ENGINEERING AND TECHNICAL SUPPORT

U.S. ARMY CORPS OF ENGINEERS  
DOE - \$12,950,000  
6/12/75 - Continuing

**OBJECTIVES** – The Corps of Engineers (CE) support program will provide DOE with engineering, technical, and contract administration support related to fossil energy demonstration plant civil and mechanical design and field construction and will expedite procurement of equipment with long lead times. This support involves technical services other than process design for facility design, construction, project management, contract management of design, and Government furnished property and/or related support for projects and locations requested by DOE. Requests for assistance are made in the form of task assignment letters describing the scope of services desired. The task letter defines the specific function requested, enumerates the contract responsibilities,



indicates the proposed location of the project, defines the funding limitations, and designates the DOE individual or office responsible for furnishing any requested DOE inputs, directions, or approvals.

**RECENT WORK AND ACCOMPLISHMENTS** – Under the current tasking letters, the Huntsville Division, CE, supported DOE in review and evaluation of contractor proposals in response to RFPs for the fuel gas, the synthetic pipeline gas, and the HYGAS demonstration plant projects. Further assistance was provided in the areas of contract negotiations and cost estimating. Upon award of the synthetic pipeline gas, i.e., CONOCO, ICGG, and the HYGAS demonstration plant projects, the Huntsville Division provided full time personnel on site to provide project management support at Foster Wheeler Energy Corporation and Dravo Corporation, the primary subcontractors for ICGG and CONOCO, respectively. Additionally, a full time project management team in Huntsville provided support in the areas of design reviews, progress assessment, cost and voucher review and approval, quality assurance planning, site requirements, configuration management, overall contract administration, scheduling, procurement, real estate, contractor instruction, environmental requirements for EIS, and preparation of draft response to contractors' letters. For the clean boiler fuel demonstration plant projects, the Huntsville Division assisted in the evaluation of the program, definition of work required to logically terminate the program, negotiation of termination, review and evaluation of cost vouchers, and participation in scheduling meetings and other contract management tasks as requested by the DOE program director. The Huntsville Division reviewed, validated, and updated to current dollars capital cost estimates for the commercial COED Facility, the commercial Fischer-Tropsch Facility, the commercial Oil-Gas Facility, the commercial Advanced Coal Liquefaction Facility, the commercial Clean Boiler Fuel Facility as designed by Coalcon, and the commercial clean boiler fuel concept as designed by Dravo Corporation. The Huntsville Division assisted in the preparation of an RFP for the atmospheric fluidized-bed boiler demonstration plant project.

**PLANS FOR THE COMING YEAR** – The Huntsville Division, CE, plans to continue supporting DOE through the Phase I design and subsequent evaluation process and final design and procurement of long lead time items of Government furnished property, site selection, environmental analysis, and contract administration for the CONOCO and the ICGG synthetic pipeline gas demonstration plants; for the conceptual commercial and demonstration plant designs of the HYGAS project; capital cost validation for various conceptual commercial plant designs; contract management assistance in the construction of the Carbondale Mining Research Center; and other support as requested by DOE.

### **SUPPORT SERVICES FOR COAL CONVERSION**

**MOUND LABORATORY**  
DOE - \$1,690,000  
3/1/76 - Continuing

**OBJECTIVES** – The objective is to provide a wide range of support services to the Coal Conversion Division to assist in the selection of contractors, and to provide technical surveillance by monitoring the efforts of the successful bidders in the Fuel Gas Demonstration Plant Program. The monitoring effort will provide assurance to DOE Program Directors that each plant built will provide sufficient data for demonstration of the technical and economic viability of the gas produced as fuel for electric power generation and/or industrial use.

**RECENT WORK AND ACCOMPLISHMENTS** – The Fuel Gas Demonstration Plant Program involves three distinct projects: a utility project directed toward electric power generation, with no restrictions on the coal feed rate; an industrial project directed toward uses such as process heat, synthesis gas for fertilizer, or chemical production, with a coal feed rate not less than 500 t/d; and a small-scale industrial project directed toward general use with a coal feed rate from 200 to 500 t/d. Second-generation noncommercial gasifier technology is required in the utility and industrial projects. First-generation commercially available gasifier technology may be used in the small-scale project. DOE defines second-generation gasifier processes as those now in a pilot plant stage of development. The three projects are divided into phases: Phase I, program development and conceptual design; Phase II, final design, procurement, and construction; and Phase III, operation and evaluation. Fifteen proposals were received in response to the RFP for this project. Mound personnel served on the Technical Advisory Committee and on the Business Management Advisory Subcommittee supporting the fuel gas Source Evaluation Board (SEB) in the analyses of these proposals. DOE's Administrator selected W.R. Grace and Memphis Light, Gas and Water Division in the industrial project and Erie Mining Company in the small-scale industrial project for negotiation of contracts. Technical support was provided for these negotiations. With the signing of these contracts, an on-site team, to be located in the office of the Architect/Engineer subcontractor selected for each contract, was formed. The primary objective of each team is to monitor the design work with respect to technology, cost, and schedule so that an "early warning signal" is provided for deviations, and that the proposed recovery plan is properly implemented to bring the work into compliance. In addition, a Resource Technology Group was formed to support the projects with such specialized disciplines as Environmental Engineering, Instrumentation, Advanced Gasifier Technology, etc. This procedure ensures that the most current technology is being applied to the design of these plants. The Resource Technology Group will also monitor the pilot plant work for second-generation technology.

**PLANS FOR THE COMING YEAR** – The three on-site teams and the Resource Technology Group will monitor Phase I work for the contracts cited above. Additional technical support is being provided to the SEB for preparation of the presentation to the Source Selection Official for the utility project. After selection, Mound will provide technical support for negotiations and after contract signing, will provide the required on-site teams for monitoring these contracts.

## COMPUTER MODELING OF COAL GASIFICATION REACTORS

### SYSTEMS, SCIENCE AND SOFTWARE

DOE - \$1,352,776

7/1/75 - 9/30/78

Principal Investigator - T. Blake

**OBJECTIVES** – General computer models will be developed, based upon continuum theories of gas-solid particle flows, and applied to the performance of fluidized-bed and entrained-flow reactors for coal gasification. It is expected that these computer models, together with experimental data and engineering experience, can be a valuable tool in the development, optimization, and scaleup of coal gasification processes. Such models, used in parametric studies, can provide a cost-effective simulation of actual experiments under gasification conditions. The influences of reactor geometry and operating conditions upon the details of the internal flows and, hence, upon the process variables that determine reactor performance, can be examined with these numerical models.

**RECENT WORK AND ACCOMPLISHMENTS** – The research effort was organized into two main task areas. One of these tasks was the continuation of computer model development for fluidized-bed gasification. The second task was the development of a computer model for entrained flow gasification. The fluidized-bed model was initially developed in the first year of the program and was applied to the study of several nonreactive flows in fluidized beds. It provides a field description of the flow processes in fluidized beds; time histories and spatial distributions of the important process variables are described explicitly by solution of the equation set. Specifically, the details of the flow such as the formation and rise of the gas bubbles, the exchange of gas between the bubbles and the emulsion region, the entrainment of solid particles in the wake of the bubble and the distribution of gas composition evolve naturally from the numerical calculations of the field within the fluidized bed. Further, the important influences of the gas phase properties and the nature of the solid particles, such as shape and particle size distribution, are included in the continuum representation. The fluidized-bed coal gasification code was modified to include species transport and kinetics for reactive fluidized-bed flows. The specific chemistry in the numerical model is representative of the processes of steam-oxygen gasification, but the numerical formulation is quite general, and a variety of heterogeneous and homogeneous reactions can be easily incorporated into the model. Test calculations have been performed to study oxidation of carbon near the distributor plate of fluidized beds. In these studies, solids convection, temperatures, and species concentrations have been calculated for some simple configurations.

The numerical model was used to study a variety of fluidized-bed flows. Some of these calculations were compared with measurements to evaluate the numerical procedures. Good agreement was found between calculated bubble size, shape and velocity and corresponding laboratory measurements. The code was also used to study distributor plate flows simulating the Synthane gasifier; these calculations of solid particle mixing, together with flow visualization experiments, are being used to understand the nature of particle convection near orifices in such distributor plates. The entrained flow coal gasification model was formulated, developed and tested. The model represents transient multidimensional flows in complicated internal geometries typical of entrained flow gasifiers. This computer code has been used to study several nonreactive gas-particle flows, including swirling flows produced by tangentially directed nozzles, simulating configurations used in combustion of pulverized coal. These calculations have involved two-dimensional geometries and are of a preliminary nature. With the development of the three-dimensional entrained flow code in the next year of this project, this study will be extended to the combustion and gasification processes of entrained flow gasifiers. The chemistry of entrained flows, which will be incorporated in the numerical model in the third year of this program, has been substantially formulated. Heterogeneous and homogeneous reaction models for combustion and gasification have been defined. A tentative approach to devolatilization, based upon relatively simplified kinetics, has been selected.

**PLANS FOR THE COMING YEAR** – In the third year of this project the chemistry and radiative transport representations will be combined into the entrained-flow code. During that third year, the

## GASIFICATION PROGRAMS AND UNIT OPERATIONS EVALUATION

FRUMERMAN ASSOCIATES INC.

DOE - \$254,142

6/76 - 12/78

**OBJECTIVES** – This program will evaluate the technical soundness, potential economic advantages, and operability of a coal gasification program; to provide recommendations for development work required to resolve engineering problems in various gasification, gas treatment, gas conversion, water treatment, and waste disposal steps of a coal gasification system. Various coal gasification processes are at different stages of development and require the solution of certain problems before they can achieve commercial acceptance. Assistance is provided to the Department of Energy (DOE) in recognizing and overcoming these technical obstacles and thereby improving the probability of success for the coal gasification program.

**RECENT WORK AND ACCOMPLISHMENTS** – Although consulting and engineering support services have been rendered to DOE under eight separate task assignments, the major effort has been concentrated on the Morgantown Energy Research Center (MERC) low-Btu gasifier and the Gasifiers In Industry Program. Together with MERC, a test program was devised using a small-scale sidestream gas cooling and cleaning system already in existence. The tests yielded data for design of a full-scale system. Conceptual design and engineering was supplied to the extent necessary so that others could perform the detailed design. Assistance was then rendered to MERC in the review of that detailed design. Efforts were next directed to the study and evaluation of various state-of-the-art processes for treatment of foul water. All were complex, costly, and required individual testing of specific effluents for the preparation of reliable designs. Attention was turned to development of a novel process to handle all liquid effluents and a conceptual design was outlined. To insure soundness of engineering and to provide constructive input, an independent review was conducted of the engineering designs for several gasifier installations in which DOE participated. These included Pike County, Kentucky (Douglas Industrial Park), University of Minnesota (Duluth Campus), and Glen-Gery (instrumentation only).

**PLANS FOR THE COMING YEAR** – Review of engineering designs will be continued for the plants named above and will be extended to include the other gasifiers in the program. Treatment and disposal of oil and water effluents and potential use of by-product tar will be evaluated for the Gasifiers In Industry Program. State-of-the-art and improved treatment and disposal technologies will be compared as to cost, possible adverse effects and operability for Gasifiers In Industry, and other gasifier applications. A number of significant design problems have arisen in the Gasifiers In Industry Program. Engineering solutions to these problems that will provide operable systems at minimum cost are being sought.

## TASK ASSIGNMENTS ON COAL CONVERSION AND UTILIZATION

GILBERT ASSOCIATES, INC.

DOE - \$1,103,227\*

3/15/76 - Continuing

**OBJECTIVES** – Gilbert provides specific engineering, scientific, and economic consulting services in support of the Department of Energy (DOE) Fossil Energy (FE) efforts to develop commercially

\*For FY 1977 only.

acceptable advanced conversion and utilization technology. Task assignments have been undertaken for the Gaseous Fuels Project Management Branch, and the Direct Combustion Branch. Additionally, work is being performed for FE's Office of Program Planning and Analysis in support of the coal conversion and utilization program. These services specifically deal with surveying and evaluation of gasification and gas cleanup systems, fluidized-bed combustion systems, and particulate sampling and measuring systems. In addition, Gilbert develops data pertinent to the commercial potential and environmental acceptability of these systems. Considerations are directed toward industrial and electric utility energy utilization for both new construction and retrofit situations. The data on these systems is utilized by DOE to help determine areas where further research and development is warranted.

**RECENT WORK AND ACCOMPLISHMENTS** — General technical support was provided to a wide range of program areas, as the following examples indicate. A technical and economic study of numerous conventional, alternative, and advanced fossil energy options for electric utility central stations was completed. This study involved solid, liquid, and gaseous fuel applications for facilities operating under base, intermediate, and peak load conditions with consideration of both new construction and retrofit situations. All of the variables were studied on a geographical basis and were assessed in terms of operating performance and costs. The fossil energy forms studied included mechanically cleaned coal, coal and oil shale derived heavy and light liquids, and low- and medium-Btu gas. An investigation of the economic effect of varying the parameters within individual unit operations of a commercial-scale high-Btu coal gasification process continued. Most of the subtasks of this comprehensive study of the anatomy of a gasification system are in the draft report stage. A study of particulate control for PFB combustion recommended a broadening of the research base and an expanded equipment development program, including filtering, cleaning and housing subsystems suitable for testing in operational pilot plants. An evaluation of the status of gas cleanup technology indicated that high-temperature high-pressure cleanup systems and components should receive development emphasis. Molecular sieves, granular beds, and alternative scrubber equipment were priorities. It was also found that gasification process modifications, such as deep bed coal injection geometry, can reduce coal gas impurity carryover without affecting total product gas yield. An ongoing study is investigating hot versus cold gas cleanup for pressurized fluidized-bed combustion. Cycle performance comparisons will be refined, major component availability will be identified, and relative costs will be compared. The high-temperature cleanup approach has received more emphasis than cold cleanup, but development difficulties have been encountered. This study will be a basis for a more detailed assessment of cleanup alternatives for the PFB program. A review on applying tentative new source performance standards (NSPS) for coal-fired steam generators to fluidized-bed combustors found that data from bench and pilot scale units demonstrate potential emissions from fluidized-bed units to be less than NSPS requirements. However, scale-up phenomena in fluidized-bed units are not defined enough to allow reasonable predictions of commercial plant emissions from the current data base. Since there are significant technological differences between fluidized-bed combustion and conventional coal-fired boiler technology, including fluidized-bed combustion under the tentative NSPS would be premature.

vanes and blades, especially when unproven hot gas cleanup components are introduced into the system. Although decoupling of the turbine was the central feature of this study, other developmental items were examined for their potential to cause a delay in testing or damage to components. An evaluation of the corrosion rates and metallurgical stability of boiler components in the 30-MW fluidized-bed boiler at Rivesville, West Virginia, was completed. Most of the tubes in the boiler were within the ASME code limit for the life of plant. Several components were designated as potential worst corrosion cases, and monitoring was recommended. An outline for a quality assurance program for future fluidized-bed boiler design, construction, and operation is also proposed. Also, for the Rivesville project, an alternative air distribution grid design was investigated. Serious erosion of the original air distribution grid would lead to maldistribution of combustion air flow and a consequent adverse effect on unit performance. The report described an erosion-resistant fallback design which could be quickly manufactured and installed. The alternative design was accepted by DOE and, after testing at MERC, the grid will be installed in the Rivesville unit in 1978. Other projects to which Gilbert provided support were Battelle agglomerating ash burner gasification, Combustion Engineering entrained flow gasification, Synthane fluidized-bed gasification, Curtiss-Wright pressurized fluidized-bed conceptual commercial plant and small gas turbine facility, Argonne pressurized fluidized-bed component test and integration unit, and the Morgantown Energy Research Center atmospheric fluidized-bed component test and integration unit. A number of unsolicited proposals to DOE were evaluated, and recommendations were made for their disposition. In several cases, such proposals held promise for important contributions to fossil energy technology.

**PLANS FOR THE COMING YEAR** – Work will continue on DOE/FE's tasks currently in progress, and new assignments will be undertaken as required by DOE.

### HOT GAS CLEANUP

#### MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$750,000

1/74 - Continuing

**OBJECTIVES** – This project, after establishing the hazardous and corrosive substances and their mechanisms in the effluent of thermally subjected coal, will deal with the treatment or removal of the undesirable materials from hot effluent while maintaining the sensible heat to increase thermal efficiency of its utilization. Undesirable materials being considered relative to end-use applications include  $H_2S$ ,  $SO_x$ ,  $NO_x$ , alkali, halogens, tars, particulates, and organics.

**RECENT WORK AND ACCOMPLISHMENTS** – A task force on hot gas cleanup has reviewed the potential applications and advantages of hot gas cleanup for contaminant control in low-Btu gasification and fluidized-bed combustion cycles. They have assessed research needs to support the development and selection of cleanup techniques for end uses of those technologies. The final report of this task force reviewed the thermodynamics of the formation of contaminants under process conditions, reviewed the chemical basis of some control technologies, and identified some research required for the development of process stream contaminant conditioning and removal techniques. Equilibrium concentrations of coal gasification and combustion product gases determined by free energy minimization of feed elements were also reported. The kinetics of iron oxide sulfidation was compared in a report on the rate of sulfidation of various metal oxide

systems. Iron oxide, while rate limited for sufficiently small pellet sizes, absorbs  $H_2S$  10-100 times faster than observed at diffusion limits for other sorbent oxides.

**PLANS FOR THE COMING YEAR** – The kinetics of  $H_2S$  sorption and regeneration of silica supported iron oxide pellets will be studied as a function of support material, temperature, and atmosphere. High-temperature iron oxide regeneration will be studied in air and  $SO_2$  atmospheres. A spectroscopic method for the measurement of reduced nitrogen concentrations in the producer gas stream will be developed. The Ames alkali monitor will be tested on the producer side stream, and a sampling train to distinguish alkali associated with half micron and larger particles from total alkali will be constructed and tested.

### HOT LOW-BTU GAS DESULFURIZATION

AIR PRODUCTS AND CHEMICALS, INC.

DOE - \$170,274

7/12/77 - 4/12/78

**OBJECTIVES** – The objective of this program is to develop information necessary to accelerate commercialization of the iron oxide process for hot gas desulfurization. Specific objectives are to determine experimentally the conditions that yield elemental sulfur during regeneration of sulfided sorbents; to perform economic analysis to determine process cost sensitivity of several variables, to determine safe conditions for regeneration of sulfided sorbents based on mathematical model and actual data and to apply APCI model to data obtained by MERC. Results of this work will generate necessary information for improving economics of the hot iron oxide desulfurization process. Removal of hydrogen sulfide from the producer gas while hot, instead of cooling it, may improve the thermal efficiency of coal conversion by up to about 5 percent.

**RECENT WORK AND ACCOMPLISHMENTS** – A new bench-scale pilot unit was designed and constructed. Analytical procedures were finalized. Economic analysis of different possible iron oxide process configuration is in progress. Mathematical analysis has been done to determine regeneration conditions that limit the maximum temperature in the bed to a safe allowable value. A mathematical model developed under a previous contract will be checked by testing reactor data from MERC.

**PLANS FOR THE COMING YEAR** – All the work described above will be completed and a final report will be prepared.

### HIGH-TEMPERATURE FUEL GAS CLEANING AND SULFUR REMOVAL WITH MOLTEN SALTS

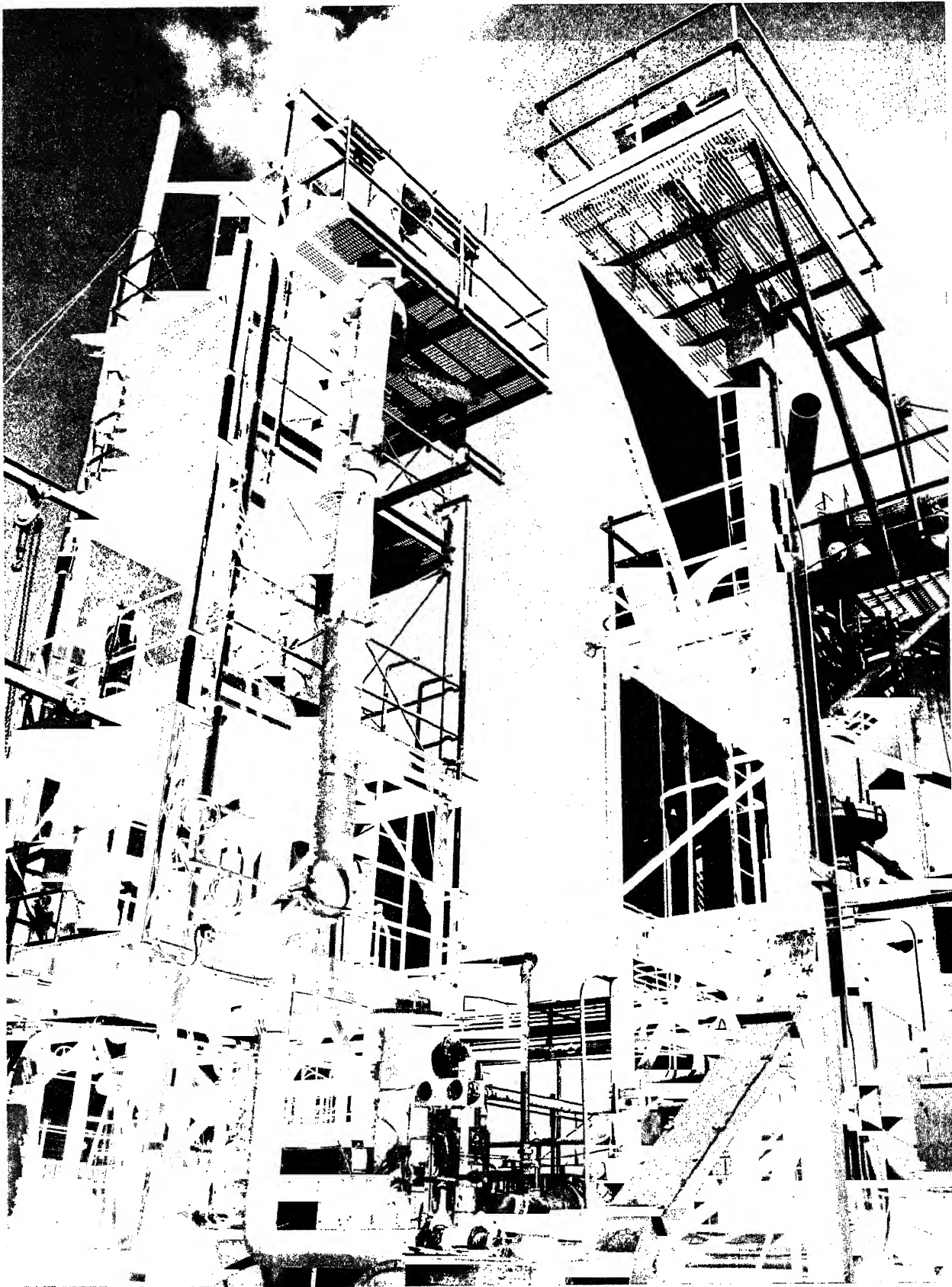
BATTELLE, PACIFIC NORTHWEST LABORATORIES

DOE - \$980,000

11/5/75 - Continuing

**OBJECTIVES** – This program will develop a molten salt scrubbing process for cleaning low-Btu gas by the removal of sulfur compounds and particles. Operating at temperatures in the range of  $1100^{\circ}$ – $1500^{\circ}$ F, the process will conserve sensible heat to maximize overall efficiency of the combined cycle while meeting gas turbine inlet gas quality requirements. The work is presently concerned with operation of a Process Development Unit (PDU) for a period sufficient to develop process parameters and to evaluate equipment components.





*Hot Fuel Gas Cleaning Process Demonstration Unit (Left) with Battelle-Northwest Gasifier (Right)*



**RECENT WORK AND ACCOMPLISHMENTS** – The combined cycle entails gasification of coal to produce low-Btu gas, hot gas cleaning, and electrical power generation by clean gas combustion and expansion through a gas turbine. Turbine exhaust gases are used to raise steam in a waste heat boiler to power a steam turbine. This combination of gas and steam cycles is a potentially more efficient method for power generation than direct combustion of coal in a conventional steam plant. The Pacific Northwest Laboratories (PNL) hot gas cleaning step preserves the sensible heat in the fuel gas and may provide additional fuel values as a result of pyrolysis of tars and oils. Previous work at the PDU scale of operation in a batch mode confirmed the ability of a venturi scrubber to achieve efficient sulfur compound removal and to circulate salt without the use of a mechanical pump. Sulfur removal efficiency ranged from 95 to 99 percent with overall sulfur removal being limited by COS which was inefficiently extracted due to the very short residence time in the single stage venturi-de-entrainer contactor. Particle removal efficiency closely approached turbine gas specifications and salt carryover was low. Only modest improvement in these parameters must be achieved to ensure production of turbine quality gas. A new PDU featuring three extraction stages and continuous regeneration of spent salt with steam-CO<sub>2</sub> has been constructed. The regenerant gas containing 30-70 percent H<sub>2</sub>S is suitable for use as feed to a Claus process. As before, one stage of extraction occurs in the vertically oriented venturi scrubber which provides the means for salt circulation. Following de-entrainment of the molten salt the gas encounters two additional extraction stages in counterflow to regenerated salt in a column containing bubble cap trays. A packed bed de-entrainer and a demister remove the last traces of salt from the cleaned gas. PDU plant shakedown tests are now in progress. The Battelle gasifier, which supplies low-Btu fuel gas, and other Battelle owned accessory equipment have been renovated and brought to operational readiness. The supporting laboratory has been equipped with analytical instrumentation for monitoring analytical plant gas streams. A detailed hazards review and safety analysis has been completed and equipment for detection of toxic CO and H<sub>2</sub>S gases has been installed and calibrated.

**PLANS FOR THE COMING YEAR** – Operation of the PDU in a series of runs to demonstrate tar and oil pyrolysis, particle (ash) removal, and sulfur removal to turbine gas specifications is planned. Precise definition of alkali metal carryover from the molten salt will be obtained. Process viability will be established by sufficiently prolonged operation of the PDU to ensure that problems with corrosion-erosion, salt stability, and equipment performance are fully defined. Continued development of the molten salt scrubbing process is contemplated. Extension of its applicability to high pressures (as well as high temperatures) will require an innovative approach. Conceptual design of such a process is planned.

## COAL CONVERSION SYSTEMS TECHNICAL DATA BOOK

INSTITUTE OF GAS TECHNOLOGY

DOE - \$1,400,132

5/1/76 - 10/31/78

quently, the Coal Conversion Systems Technical Data Book project was created by OCR in October 1974 to begin compiling the necessary data for coal conversion systems. The objective of this work is to provide a single comprehensive source of data for the design and evaluation of coal conversion systems. The compilation will include up-to-date information for the research, development, design, engineering, construction, and operation of coal conversion processes, process facilities, and plants. Other concurrent objectives are to identify those areas where data are needed but unavailable and to suggest research programs for providing required data.

**RECENT WORK AND ACCOMPLISHMENTS** – IGT is collecting, evaluating, and compiling reliable data in a form useful to scientists, engineers, designers, and operators engaged in coal conversion work. The data sources include work done by DOE contractors in coal conversion, pertinent data and correlations from the published literature, pertinent data and correlations from unpublished sources within Government, industry, and private sectors, and development of new correlations where applicable, to fill gaps in existing data. During the period May 1, 1976, to October 31, 1977, the following areas were partly covered under the 10 project tasks. Task I, Properties of Process Materials, covered vapor-liquid equilibrium with respect to solubility of gases in hydrocarbon liquids, solubility of gases in water, solubility of oils in water (one liquid phase), solubility of water in oils (one liquid phase), and water-oil systems (two liquid phases). Other task areas included: vapor-liquid equilibria in the  $\text{NH}_3\text{-H}_2\text{S-CO}_2\text{-H}_2\text{O}$  system, particle density of coal and char, bulk density of coal, calorific value of coal, size consist of coal from various mines, influence of mining techniques on size consist and washability of coal, trace elements in coal, properties of coal liquids, and properties of pure components including phenol, O-M-P cresols, and four-ringed aromatic hydrocarbons. Task II, Solids Storage, Handling, Preparation, Pretreatment, and Feeding, covered the following areas: coal sampling, storage, and conveyance; coal preparation; sulfur reduction in coal by washing; and pulverization and storage of solvent-refined coal. Task III, Conversion Fundamentals, covered the areas of steam-air gasification of char in a fluidized bed, pyrolysis, the fate of trace metals in coal conversion processes, fluidized-bed combustion: sulfur retention capability of limestone and dolomite, and equilibrium constants of coal conversion reactions. Task IV, Design Procedures, covered the areas of fluidization, solids transport, coal solvent slurry heating, and heat transfer in fluidized beds. Specifically, the area of fluidization covered revision of the empirical bed-expansion correlation, solids mixing in fluidized beds, initial bubble-diameter correlations, bed-expansion correlations from two-phase theory and various bubble-growth correlations, and fluidized-bed combustion: air-distributor design. The area of solids transport covered generalized phase diagrams for two-phase, fluid-particle systems; evaluation of choking velocity correlations; pressure-drop correlations in vertical, upflow solids transport in lean-phase; gas-solids upflow in inclined pipes; gas-solids downflow: standpipe design in fluidized-flow and packed-bed regime; and gas-solids flow in horizontal pipes: saltation velocity correlations. The area of coal solvent slurry heating covered the estimation of physical and transport properties of slurries, mathematical model for slurry viscosity behavior in preheaters, heat transfer correlations, slurry classification and pressure-drop calculations, and comparison with experimental data on SRC, Wilsonville preheater. Task V, Supporting Processes, covered environmental regulations, trace element disposition in coal combustion and occurrence in water, and stack-gas cleaning processes. Task VII, Materials of Construction, covered experience of the HYGAS pilot plant, outline of the section on properties of metals and alloys, and evaluation of performance of refractory materials in condensing the acid-gas atmosphere.

**PLANS FOR THE COMING YEAR** — Plans include continuation of work in collecting properties of process materials; increased emphasis on solids handling and preparation; continuation of work in gasification and fluidized-bed combustion fundamentals; initiation of work in liquefaction fundamentals; continuation of work in solids transport and heat transfer; start-up of work in phase separations, vapor-liquid transport; irreversible work analysis, and hydroprocessing of coal liquids; and increased emphasis on environmental control and materials of construction. Plans also call for initiating some work in process economics and preparing process flow sheets for presentation in the Data Book. The first volume of the Data Book will be published in August, 1978. It will deal with the physical and chemical properties of coal, char, and ash. A preliminary index for the material to be published has been prepared. The first unit of additions and revisions (a 6-month spacing will be used for issuing addition/revision supplements) will also be issued. Because new data are continually being generated by various coal conversion R&D activities, this effort is a continuing task.

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### PUBLICATIONS

- Ellman, R.C., and Schobert, H.H. "Pilot Plant Operation of a Fixed-Bed Slagging Gasifier." Presented at the 173rd National Meeting of the American Chemical Society, New Orleans, La., Mar. 1977.
- Kornosky, R.M. et al. "Gasification of Iowa Coal in the SYNTHANE PDU Gasifier." *Preprints*, 174th Meeting of the American Chemical Society, Fuel Chemistry Division, 22, No. 2: 197-205 (Mar. 1977).
- Macko, J.E. et al. "Instrumentation, Development, and Application at the Westinghouse Coal Gasification PDU." Presented at the 1977 Symposium on Plant Instrumentation, Chicago, Ill., July 1977.
- Margaritis, P.J. et al. "Operation of the Westinghouse Fluidized Bed Devolatilizer With a Variety of Coal Feedstocks." Presented at the 173rd National Meeting of the American Chemical Society, New Orleans, La., Mar. 1977.
- Miles, J.M. "More Tests to Expand BI-GAS Pilot Plant Experience." *Oil and Gas Journal*, Aug. 1, 1977, pp. 55-58.
- Pukanic, G.W.; Haynes, W.P.; and Schehl, R.R. "Statistical Analysis of Linear Programming Model for Illinois No. 6 Coal Gasification Data From the SYNTHANE Process." Pittsburgh Energy Research Center. PERC/RI-77/1 (Jan. 1977).
- Rath, L.K. et al. "Operation of the Westinghouse Coal Gasification PDU." Presented at the Fourth National Conference on Energy and the Environment, Cincinnati, Ohio, Oct. 1976.
- Saroff, L. et al. "Entrained Pretreatment and Coal Transport." *Chemical Engineering Progress* 3: 172-79 (1977).

# ***ADVANCED POWER SYSTEMS***

The Advanced Power Systems activity addresses baseload electrical generation by central station utilities. Coal and coal-derived low-Btu gases are projected to be the predominant fossil energy resource for electric utilities in the future, and the objective is to provide technology for increasing the utilization of this abundant fuel resource to offset the use of critical oil and gas in the utility market. The open-cycle gas turbines developed in the United States have been primarily for military and civilian jet aircraft. The United States has also developed heavy-duty, long-life, open-cycle turbines for the utility and industrial markets; however, both aircraft and utility gas turbines have been designed to burn clean fuels to achieve acceptable durability in baseload service and meet emissions standards.

Integrated coal conversion and utilization systems have shown to be a most promising option for efficiently using coal in an economic and environmentally acceptable manner. Specifically, an integrated high-temperature gas turbine/low-Btu coal gasification combined-cycle plant holds significant promise for achieving the high efficiency needed to offset the energy losses in cleaning and gasifying coal so that electricity can be produced at a comparable cost with oil-fired steam and nuclear baseload systems. Such a system would use materials, internal cooling, and components designed specifically for high reliability utility service and reject heat to a heat recovery boiler to generate additional steam turbine power. This system concept offers potentially lower capital cost, shortened construction time, and higher efficiency than a conventional high-pressure steam power plant. The program is to establish a technological base for use in developing a combined cycle or similar system. A gas turbine with a high turbine inlet temperature (over 2500°F) burning coal-derived fuels is the key component for this system. The critical technical problem that is being solved to commercialize such a plant is cooling of the high-temperature turbine components.

Major potential economic advantages are offered by integrated coal conversion and utilization systems as a result of possible lower capital costs of high-performance combined-cycle turbine systems, and from their attractive coalpile-to-busbar efficiency. Additional benefits include lower environmental impact (reduced consumptive water use, clean flue gas), reduced fuel usage resulting from higher efficiency, and reduced usage of premium-priced synthetic/natural gaseous and liquid fuels.

Closed-cycle power systems integrated with a primary heater or a fluidized-bed combustor represent another environmentally promising approach for utilizing coal directly in utility baseload service. Although with current technology, this option is potentially less efficient than the advanced combined-cycle gasification plant, closed-cycle systems can be considerably more efficient than conventional coal-fired boiler systems particularly when a closed-cycle system is used in a topping cycle mode to extract currently unused energy available in the combustion products at temperatures above the 1050°F steam temperatures of conventional systems. The critical component limiting closed-cycle power systems is the primary heater. Closed-cycle systems are undergoing assessment, supported by some experimental efforts.

**PLANS FOR THE COMING YEAR** — Plans include continuation of work in collecting properties of process materials; increased emphasis on solids handling and preparation; continuation of work in gasification and fluidized-bed combustion fundamentals; initiation of work in liquefaction fundamentals; continuation of work in solids transport and heat transfer; start-up of work in phase separations, vapor-liquid transport; irreversible work analysis, and hydroprocessing of coal liquids; and increased emphasis on environmental control and materials of construction. Plans also call for initiating some work in process economics and preparing process flow sheets for presentation in the Data Book. The first volume of the Data Book will be published in August, 1978. It will deal with the physical and chemical properties of coal, char, and ash. A preliminary index for the material to be published has been prepared. The first unit of additions and revisions (a 6-month spacing will be used for issuing addition/revision supplements) will also be issued. Because new data are continually being generated by various coal conversion R&D activities, this effort is a continuing task.

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## HIGH-TEMPERATURE TURBINE TECHNOLOGY PROGRAM

CURTISS-WRIGHT CORPORATION  
DOE - \$29,384,000  
5/28/76 - 1/31/80

**OBJECTIVES** – This program will develop, to a technology readiness level, the turbine subsection of a gas turbine for a combined cycle electric generating plant using low-Btu coal gas fuel and high turbine inlet temperatures (2600° - 3000°F) to increase gas turbine efficiency and compensate for the cost of producing coal derived fuel gas. The program is being conducted in three phases, over a 6-year time span. Phase 1, completed in 1977, defined the systems and development efforts required to complete the program. Phase 2, which is currently funded, consists of technology testing and support studies that will obtain the technical data necessary to complete the subsystem design to be verification tested in Phase 3. Successful completion of this program will result in a high efficiency, advanced-cycle power system capable of operating on coal derived fuel in an environmentally acceptable manner. The availability of such a system will accelerate the re-introduction of coal as a prime fuel for electric power generation.

**RECENT WORK AND ACCOMPLISHMENTS** – Phase 1, Program and System Definition, was completed with the publication of nine technical topical reports and a final report. Major accomplishments of this phase are as follows: (1) a conceptual design of an integrated low-Btu coal-gas fueled, 750-Mw commercial power generation plant, (2) a conceptual design of a 665-Mw commercial power generation plant fueled by liquified coal, (3) preliminary designs of combined cycle systems using coal derived fuels, (4) preliminary designs for a 3000°F, transpiration air cooled turbine subsystem, and a 3000°F steam-cooled turbine back-up system, (5) identification of forward R&D requirements including plans for Phase 2 turbine component testing and Phase 3 gas-generator verification testing, and (6) designs for three different conceptual low-Btu coal gas combustors including plans for an independent verification combustor test program.

Phase 2, Technology Testing and Test Support Studies, is primarily concerned with the completion of nine major tests to be conducted on combustor sector, turbine stator cascades, and full-scale rotating engines. Fuels to be used consist of distillate jet fuel, a distillate fuel doped with particulate and alkali metal salts to simulate a coal derived gas, synthesized low-Btu gas, and actual low-Btu coal-gas at a gasifier site. The major test plan is supported by analytical studies in heat transfer, aerodynamics, blade cooling, and performance. Metallurgical support and experimental studies include erosion, hot-corrosion/erosion, mechanical property evaluation, electron beam welding, diffusion bonding, evaluation of thermal barrier coatings, and thermal fatigue studies. The program phase is divided into four major tasks: (1) design and construction of test equipment and facilities, (2) design and fabrication of test equipment, (3) technology testing, and (4) analytical and metallurgical support studies. The Phase 2 program has been completely scheduled and individual significant milestones identified. At this time, design and analysis activity is in progress on test equipment and components. Facility preparation is continuing and long lead procurement has been initiated.

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combustor sector and a high-pressure turbine vane cascade. Designs and major procurement will be completed for combustor sector and cascade testing on synthesized low-Btu gas fuel. Designs will also be completed and procurement initiated for testing of (1) a high-pressure engine at the gasifier site on low-Btu coal-gas fuel and (2) a combustor sector for the Phase 3 verification test engine. Procurement will be completed and testing initiated on all elements in the metallurgical support program.

## **HIGH-TEMPERATURE TURBINE TECHNOLOGY PROGRAM**

**GENERAL ELECTRIC COMPANY, GAS TURBINE DIVISION**

**DOE - \$34,300,000**

**5/28/76 - 8/1/81**

**OBJECTIVES** – The High-Temperature Turbine Technology (HTTT) Program plans to bring to technology readiness within 6 years a large utility size high-temperature gas turbine for use in a combined cycle system. The turbine will use coal-derived fuel at a firing temperature of 2600°F with growth capability to 3000°F, while significantly increasing overall plant efficiency. Uncertainties in petroleum availability and cost have created an urgent need to utilize abundant reserves of domestic coal for the production of substitute fuels. Converting coal to clean-burning fuels, however, requires the expenditure of significant amounts of energy, thereby reducing the overall fuel to shaft power or electrical power efficiency. One promising method for increasing utility baseload plant efficiency using coal-derived fuels is through use of an advanced design, combined cycle generating system. Reaching this level of efficiency while fulfilling other demanding applications with petroleum fuels. Studies indicate that potential coal to busbar efficiencies of 41 percent and above are possible in another similar type integrated, advanced low-Btu gasifier combined cycle generating system. Reaching this level of efficiency while fulfilling other demanding baseload operating requirements, however, is predicated on achieving two difficult technological objectives: first, turbine inlet temperatures must be increased to values of 2600°F and above while limiting the concomitant energy losses (e.g., for component cooling) and, second, the gas turbine must be adapted to the coal-fuel combustion environment. The program has been divided into three phases: Phase I, program and system definition; Phase II, technology testing and test support studies; and Phase III, technology readiness verification test program. The work scope for the overall three phases of the DOE-HTTT advanced program encompasses the design, development, manufacture, and test of a turbine subsystem. Phase I was completed and Phase II commenced August 1, 1977. Phase III is not scheduled to commence until at least 1980. The objectives of Phase II are to (1) perform component and subsystem technology testing in critical areas; (2) conduct concurrent design and planning efforts addressed toward the completion of the technology readiness vehicle (TRV) preliminary design, as well as plans and schedules for the manufacture and testing of the TRV; and (3) update the Phase I initiated turbine prime reference design (PRD) and those coal-fuel burning combined cycle power plants featuring it.

**RECENT WORK AND ACCOMPLISHMENTS** – Phase I has been completed. Overall plant design



descriptions, cycle calculations, site plan drawings, equipment lists, operating modes, trade-offs, costs, failure mode analysis, and other data were developed for each of the above configurations. The coal pile to busbar efficiency (HHV) for advanced fixed-bed gasification system is 41.42 percent, for the entrained-bed gasification system is 41.06 percent, and for the coal-derived liquid system is 36.20.\* A preliminary design for the water-cooled machine conceived for the Phase I plant was completed including aerodynamics, heat transfer, mechanical design, materials, and manufacturing considerations. The turbine nozzles are cooled by pressurized (1250 psia) water in a closed circuit which is integrated in the steam cycle. The buckets are cooled by water in an unpressurized open circuit, allowing two-thirds of the water to vaporize so as to reduce coolant flow requirements. Steam is discharged from the bucket tips. Steam cooling and air cooling were also studied. Water cooling was selected because it offers the best overall efficiency, growth capability, and fuel flexibility. The increased heat transfer capability of water allows blading metal surface temperatures to be kept below 1000°F to reduce hot corrosion. Also, film cooling holes are not required in the blading surface so that plugging due to dirty fuels is eliminated. Materials and process investigations were also performed during Phase I and more will be completed in Phase II, including the evaluation of composite material fabrication techniques with powder metals. Three preliminary combustor designs were completed during Phase I: sectoral, annular, and cylindrical with a water-cooled transition piece. The sectoral combustor was selected because it incorporates advanced aircraft engine combustor technology with circular combustor maintainability. Twenty air-cooled sectoral combustors are required per gas turbine. Phase II and Phase III program plans and costs were also developed.

Under Phase II work, procurement of a hot gas path development test stand has been initiated, and bids have been received for its construction. The test stand is designed to accommodate full pressure and temperature testing of the combustor as well as the state 1 and stage 2 nozzles for which design has been initiated. Design requirements and operating conditions are being developed for the construction and testing of a low-temperature gas cleanup system to be operated with the GEGAS fixed-bed gasifier to provide low-Btu gas for small-scale combustion and nozzle testing. Planning and design efforts are also underway for various heat transfer and aerodynamic tests to be conducted in Phase II. Further materials and process work has been initiated in Phase II. Some materials, notably copper alloys, have been procured for evaluation tests. Programs are being finalized for process developments required for all component designs. Also, programs for nondestructive testing techniques, particle erosion, and stress corrosion cracking are also being established. Extensive testing, including rupture, creep, and low cycle fatigue, of the base and composite materials will be carried out in Phase II.

**PLANS FOR THE COMING YEAR** – Design and construction of test support equipment for most of the Phase II tests will be completed. Design of the test components and specimens will be completed and specimen fabrication will get underway. Some heat transfer testing will start in 1978 and carry over to 1979. Work will continue on the design of the TRV components. Updates of the OPDD's developed in Phase I will reflect changes evolving from Phase II activities to the water-cooled turbine design and the fuels plant.

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\*based on a coal liquefaction process efficiency of 67 percent. Fuel to busbar efficiency is 48.66 percent.

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## HIGH-TEMPERATURE TURBINE TECHNOLOGY PROGRAM

WESTINGHOUSE ELECTRIC CORPORATION

DOE - \$3,016,235

6/1/76 - 7/15/77

**OBJECTIVES** – The HTTT Program will bring to the point of technology readiness the key advances in combustion turbine technology required for the development of a high-temperature engine which would burn coal-derived fuels. This engine is intended primarily for application in an integrated coal gasification combined cycle power plant. Comprehensive studies, such as the DOE sponsored Energy Conversion Alternatives Study (ECAS), have shown this type of plant to be one of the more promising concepts for achieving significant and relatively near-term gains in efficient production of electricity while using high-sulfur coal in an environmentally acceptable manner. The advanced design engine would also provide a highly efficient conversion device for the use of coal-derived liquid fuels. The projected cost for clean gaseous and liquid fuels produced from coal dictates that such highly efficient conversion systems will be necessary to justify their use to electric power generation. The high-temperature combined cycle, operating with coal gasification systems now under development, has a projected energy savings of 10-15 percent compared to coal-fired steam plants with stack gas cleanup. The key technology goals of the HTTT Program are: (1) increasing the turbine (expander) temperature capability to 2600°F and above, utilizing an advanced design approach that minimizes the energy losses associated with component cooling, and (2) adapting the advanced high-temperature design approach to long-life reliable baseload electric utility operation in a coal-derived fuel combustion environment.

**RECENT WORK AND ACCOMPLISHMENTS** – The work completed under this contract covered Phase I, program and system definition. Extensive preliminary design studies and an assessment of the state-of-the-art were performed. The advanced research and technology development requirements were identified, and a comprehensive work plan for the Phase II and Phase III effort required to achieve the HTTT Program goals was developed. Ten topical reports, covering the main areas of technical effort and program planning, have been submitted. System design studies resulted in the development of a reference design and overall plant design descriptions (OPDD's) for an integrated coal gasification combined cycle power plant and for a combined cycle burning coal-derived liquid fuel. The coal-gas cycle was based on the use of a fluid-bed gasification process and a commercially available cold gas cleanup train. Plant efficiencies of 41 percent and 49 percent were calculated for the gasification (coal to busbar) and liquid fueled plants, respectively. The OPDD's provide system-level specifications for the construction of these plants. The Westinghouse high-temperature turbine design is based on the use of the "Lamilloy" advanced design cooling concept developed by the Detroit Diesel Allison (DDA) Division of General Motors, a major subcontractor in the project. Lamilloy, which features transpiration cooling, has been successfully demonstrated by DDA in aircraft engines at temperatures well in excess of the baseline 2600°F HTTT Program goal. Preliminary design definition of the critical high-temperature turbine components and of the overall reference design turbine assembly has been completed. Similar preliminary design work was performed for a one-third scale model of the reference design for use in the conduct of the Phase III, technology verification testing. This test vehicle size was selected to be compatible with coal gasifiers that are expected to be operational in the required timeframe. The full-scale engine will have power output of approximately 125 Mw at the baseline 2600°F turbine inlet temperature.

**PLANS FOR THE COMING YEAR** – Work under this contract was completed in FY 1977.

## HIGH-TEMPERATURE TURBINE TECHNOLOGY PROGRAM

UNITED TECHNOLOGIES CORPORATION

DOE - \$2,055,000

5/28/76 - Continuing

**OBJECTIVES** — This program is concerned with development of the technology of a 100 megawatt and larger size industrial, high temperature, open cycle gas turbine to a "technology readiness" condition within a 6- to 10-year period. The high-temperature gas turbine engine (turbo compressor) is to be capable of operating with a turbine inlet temperature of at least 2600°F (1430°C). This gas turbine is intended for use in a combined cycle plant and will utilize coal-derived fuel and be capable of base load operation by the electric utilities. A combined cycle plant consists of an open cycle gas turbine exhausting into a waste heat boiler which in turn supplies steam to a steam turbine. Both turbines drive electric generators which in turn produce electric power. The total program is divided into three separate phases: program and system definition, technology testing and test support studies, and technology readiness verification test program. The work undertaken during this report period is for the first phase only.

**RECENT WORK AND ACCOMPLISHMENTS** — Preliminary designs and technology acquisition program plans were finalized. Overall plant design has been conceptually defined for operation with gaseous fuel, or base load utilization. The plant arrangement consists of a combined cycle as the basic power system, operating in conjunction with an advanced coal gasification system. For this application, a molten salt gasification process is used for the production of a sulfur free, low-Btu gaseous fuel. The overall system (four gas turbines and one steam turbine) has the capability of producing a net output of 955 Mw and, as defined for base load utilization, it would operate in excess of 8100 hours annually and have an assumed life of 30 years. Baseline combined cycle work involved defining the thermodynamic cycle of a gas turbine engine operating at a temperature of 2600°F, a steam bottoming cycle, and a coal gasification system. The maximum metal temperature for the water-cooled turbine vanes was defined at 1500°F. The type of steam system selected for both systems consisted of a single pressure reheat cycle operating at a pressure of 2400 psia and reheat and superheat temperatures of 950°F. The nearly complete carbon utilization, no steam consumption for many coals, in situ desulfurization, and no formation of tars or appreciable amounts of potential pollutants such as NO<sub>x</sub> and NH<sub>3</sub> all contribute to the attractiveness of the molten salt gasifier. For the turbine subsystem design, preliminary reference and alternate combustion turbine designs were prepared for 2600°F combustor exit temperature with growth capability to 3000°F. In the primary design, stationary components such as vanes and outer air seals are cooled with subcooled boiling water. Subcooled boiling water is defined as high-pressure water (1100 psi) flowing in a passage where the bulk fluid temperature is below the boiling point of the water and the passage wall is above the boiling point of the water. Rotating components such as turbine blades and disks use precooled air as the cooling medium. In contrast to the primary turbine, the alternate concept employs water-cooled blades. Three preliminary low-Btu combustor designs were prepared. All employ a premix combustion concept in an annular liner. Plans were established for acquisition of the technology required by the turbine and combustor designs.

**PLANS FOR THE COMING YEAR** — United Technologies will not perform Phase II work as defined by the technology acquisition program plan, above. However, certain high-priority investigations of interest to DOE and United will be performed at no additional cost to the government under a pending contract modification.

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## COMBINED CYCLE RESEARCH PROGRAM

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

DOE - \$703,550

7/1/76 - 6/30/79

**OBJECTIVES** – The Combined Cycle Research Program is aimed at providing essential data for the future development of advanced gas turbine/steam turbine combined cycle power plants. The major benefits of this combined cycle system are the potential attainment, at relatively low risk and development cost, of high efficiencies while maintaining pollutant emissions within environmental specifications. This three-year program deals with critical, highly-coupled problems related to combined cycles: gas turbine cooling, materials corrosion, and fuel gas desulfurization. The result of research in these areas will provide needed data for gas turbine component design and thus serve as a necessary complement to the DOE High-Temperature Turbine Technology Program. This effort will also supply corrosion data for other metal and ceramic components and necessary information concerning desulfurization at high temperature, as well as providing accurate experimental data for input into, and verification of, the various computer simulator models under development.

**RECENT WORK AND ACCOMPLISHMENTS** – Work Effort I, gas turbine heat transfer and cooling research, includes testing of two-dimensional airfoil cascade representative of the nozzle of a large, high-temperature industrial gas turbine. The testing is to be done at simulated gas/metal temperature ratios encountered in advanced combined cycle systems to determine localized heat transfer. A facility to investigate water cooling of stationary and rotating turbine hardware is being built and tested. Stationary tube burnout tests have been conducted in this facility. Alternative approaches for cooling rotating blades have been investigated. A detailed analysis of property variations for boiling two phase flow in rotating channels has been completed, as well as a study of critical water film thickness at channel entrance and flow stability. Work Effort II, gas turbine materials corrosion research, includes use of an electrochemical cell to study the attack of sodium sulfate on the protective oxide coatings of turbine hardware. Since there is evidence that tungsten and molybdenum in a super alloy may influence the hot corrosion process, the effect on corrosion attack of adding tungsten oxide to sodium sulfate is also being investigated. The possible role in hot corrosion played by wetting of the protective oxide by liquids such as sodium sulfate is also being studied. An experimental apparatus was set up to measure the effects of composition, temperature, and pressure on the surface tension of sodium sulfate. Preliminary work was started on the kinetic aspects of the processes by which a protective oxide is dissolved by liquids such as sodium sulfate. An apparatus has been built to study the rate at which a ceramic material dissolves in molten sodium sulfate at high temperatures in equilibrium with gas mixtures of known composition. Work Effort III, fuel gas high-temperature desulfurization research, includes testing of a pressurized thermogravimetric analyzer facility with all auxiliary equipment. Synthetic dolomites of several calcium/magnesium ratios were successfully prepared and subjected to preliminary desulfurization and regeneration experiments. Surface area and porosity of dolomite were determined using a newly acquired mercury intrusion porosimeter. An experimental apparatus to measure effective diffusion coefficients and tortuosity factors in synthetic dolomite was constructed.

**PLANS FOR THE COMING YEAR** – The turbine nozzle cascade will be extensively tested over a range of operating conditions corresponding to both air and water-cooled engine designs to

determine the effect of gas/metal temperature ratio on airfoil heat transfer. The rotating water cooling facility will be used to determine heat transfer, burn-out, and stability criteria for water-cooled rotating turbine blades. A program to investigate the properties of thin water films in turbine blades will be initiated. The experimental program to assess the thermodynamic, kinetic, and solubility aspects of hot corrosion will continue. The determination of the effect of calcium/magnesium ratio in dolomite on high-temperature desulfurization and regeneration will continue to be investigated.

### **CLOSED GAS TURBINE HEATER PROGRAM**

**AIRESEARCH MANUFACTURING COMPANY OF ARIZONA**

**DOE - \$956,708**

**4/1/77 - 3/31/78**

**OBJECTIVES** – This program is designed to advance high-temperature closed gas turbine power conversion systems to a point of technological readiness for use in coal fired public utility power conversion plants. The initial contract addresses two specific problem areas: (1) The identification and analysis of system concepts which offer highest overall plant efficiency consistent with lowest cost of electricity from coal pile to bus bar; and (2) the identification and preliminary design of combustor/heat exchanger concepts compatible for use as the cycle gas primary heater for those plant systems. System cycle analyses and parametric studies will be performed to evaluate entire closed gas turbine utility power plants, with and without Rankine bottoming cycles, with emphasis on lowest cost of electricity. Contract completion will result in utility plant layouts, component definitions, performance, technology assessment, and costs for four heater systems. Two heater systems will be based on 1550°F closed gas turbine inlet temperature using metallic materials, and two will be based on 1750°F and higher using nonmetallic materials.

**RECENT WORK AND ACCOMPLISHMENTS** – Design and analysis computer programs for direct coal fired combustor/heat exchanger systems equipped with radiant/convective furnaces with downstream emission control apparatus and atmospheric fluidized beds operating with sulfur sorbent addition have been completed. The program provided for the significant design and operating parameters for the combustor/heat exchanger components and auxiliaries (fans, coal handling, etc.) and the cost and erection estimates for these. Computer programs have been assembled that design the closed gas turbine system using air or helium as the working fluid and, where used, various Rankine bottoming cycles. These programs are used in concert to project overall utility power plant performance, the cost of all of the plant components and the cost of electricity, both for initial operation and over 30 years of life. More than 7500 complete plant systems have been designed, costed and evaluated. Air proved to be the superior working fluid for the closed gas turbine.

**PLANS FOR THE COMING YEAR** – The selected systems will be studied in greater detail in order to identify off-design conditions, technological risks, and required development for the next phase of the program.

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## ALKALI METAL VAPOR TOPPING CYCLE

OAK RIDGE NATIONAL LABORATORY

DOE - \$1,380,000

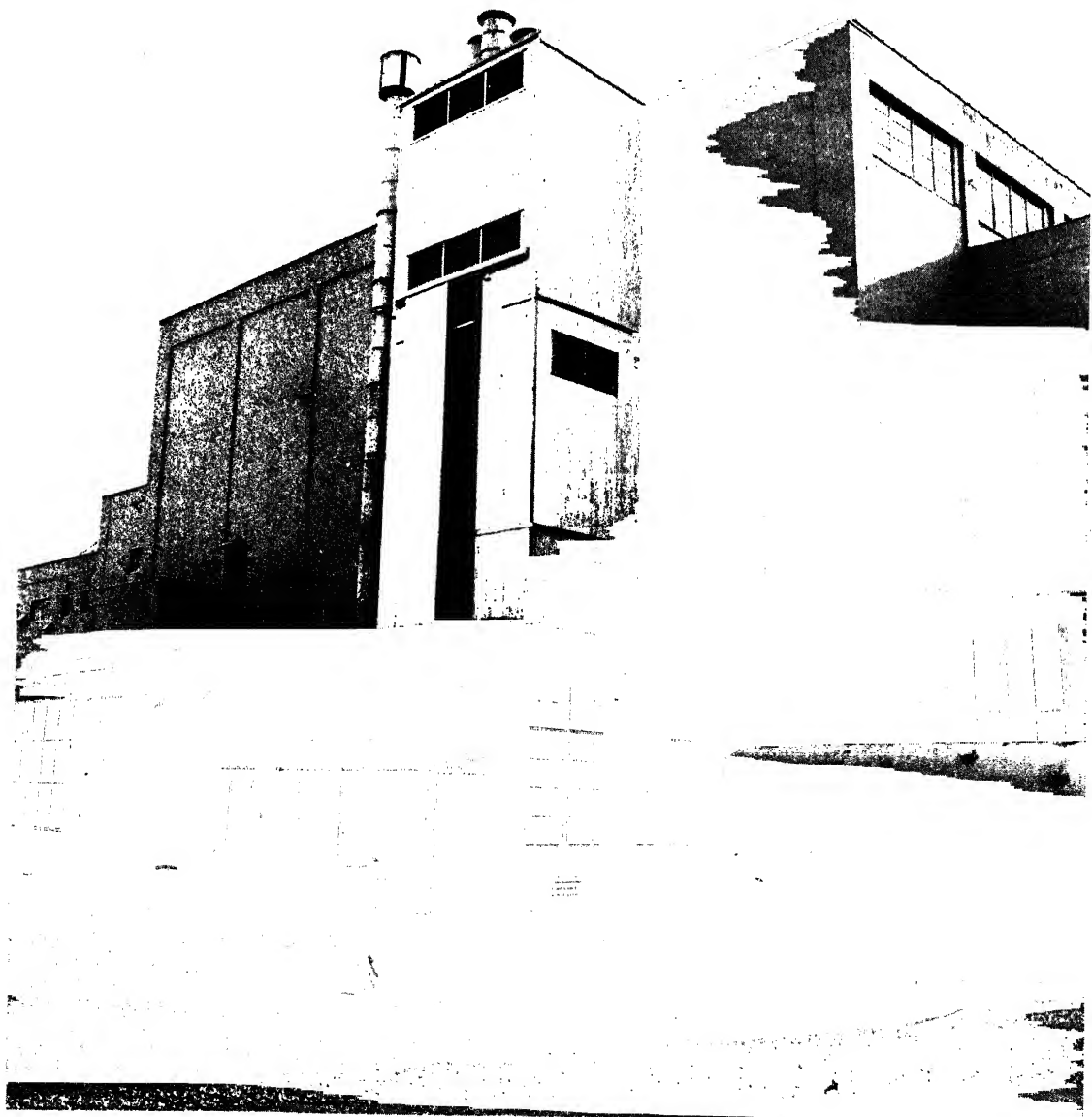
1976 - Continuing

**OBJECTIVES** — A potassium boiler tube bundle and gas-fired burner module will be built and tested to establish data on performance, cost, and reliability. This information will be the basis for design of an alkali metal boiler using potassium or cesium and operating in a coal-fired fluidized-bed furnace. An important step to reduce national fuel consumption is to increase the efficiency of thermodynamic cycles. A major factor that prevented fuel consumption from increasing more rapidly than it otherwise would have during the past century has been an increase in the thermal efficiency of steam plants by about a factor of 8. Unfortunately, around 1950 the upper practicable temperature limit for the steam cycle was reached, namely, about 538°C (1000°F). To reach higher thermal efficiencies, it will be necessary to go to higher temperatures; in turn, this implies the use of other working fluids. A leading candidate for an advanced thermodynamic cycle is a potassium or cesium vapor cycle. The approach being investigated by ORNL entails raising the peak temperature of the thermodynamic cycle by employing a potassium vapor Rankine topping cycle that operates with a turbine inlet temperature of 815°C to 870°C (1500°F to 1600°F) in conjunction with a conventional steam cycle having a turbine inlet temperature of about 538°C (1000°F) by transferring the waste heat rejected from the condensing potassium vapor to the boiling water of the steam cycle. The combined cycle is similar to the mercury-steam binary vapor cycle employed in seven U.S. plants built between 1925 and 1948; however, the operating temperature of these plants was limited to 483°C (900°F) because at higher temperatures, severe corrosion of all types of steel by mercury made operation impractical. The 438°C (900°F) mercury turbine inlet temperature was higher than the steam turbine inlet temperature in plants of the 1920's; by the late 1930's this temperature was surpassed by new steam plants whose higher temperatures and thermal efficiencies made the mercury vapor system obsolete.

**RECENT WORK AND ACCOMPLISHMENTS** — The boiler test with water was completed, yielding information in the following areas: nucleator argon distribution, boiler tube heat load distribution and boiling stability, boiler temperature distribution, and vapor separator performance. The results of the argon distribution tests indicate that the argon flow rate in all of the boiler tubes is adequate to provide nucleation promotion for stable boiling of potassium. Measurements of the heat load on individual boiler tubes with a calorimeter show that the outer bank of tubes has an average heat load of about 67 percent of the average for the inner bank, and the variation for individual tubes is about  $\pm 10$  percent from the average for each bank. Steady boiling in the outer row of tubes was observed for burner power levels of  $2.8 \times 10^6$  Btu/hr or higher. Temperature data from thermocouples on the boiler tubes during startup indicate a peak temperature difference of 25°C (45°F) between tube temperatures in the inner and outer banks, with the temperature of the liquid downcomer about halfway between these values. Tube temperatures measured under steady boiling conditions indicate that all of the tubes in the bundle are operating at temperatures within a few degrees of each other. On the basis of these results, no problems from thermal stresses are expected. The combustion gas temperature measurements indicate a fairly uniform flue gas distribution and good heat transfer to the boiler tubes. The vapor separator test yielded a steam exit quality of greater than 99.9 percent, which was better than the design goal of 99 percent.

The design and fabrication of components for the potassium test system and the installation of the system were completed this year. The potassium piping system was pressure and leak tested and found to be leak proof. The checkout of the instruments and controls was begun and nearly completed. The fill and drain tank was filled with potassium in preparation for initial shakedown testing of the boiler.

***PLANS FOR THE COMING YEAR*** – The potassium test program will be initiated and is planned to be completed by July 1978. The program calls for shakedown testing, performance tests up to a power level of  $4 \times 10^6$  Btu/hr and an endurance test of 1000-2000 hours. The test results will be analyzed, and a final report issued in September 1978.



*Potassium Boiler Test Facility*



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**CRITICAL RESEARCH AND ADVANCED TECHNOLOGY SUPPORT**  
**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION, LEWIS RESEARCH CENTER**  
**DOE - \$1,750,000\***  
**7/77 - Continuing**

**OBJECTIVES** – This project is designed to provide Federal technical support for the DOE gas turbine program to accelerate implementation of advanced systems. Advanced gas turbine systems must be capable of firing coal-derived fuels in an environmentally acceptable manner if they are to be competitive with other advanced power generation systems. Combustor concepts will be studied to minimize NO<sub>x</sub> emissions from fuels high in fuel-bound nitrogen. Experimental studies of hot corrosion of turbine materials will be performed to provide a data base for a corrosion life prediction model for contaminated fuels. Experimental and analytical studies will be performed on advanced concepts, such as thermal barrier coatings, to evaluate their potential with coal-derived fuels.

**RECENT WORK AND ACCOMPLISHMENTS** – This project was initiated on June 30, 1977, and an annual operating plan was prepared in July covering the balance of FY 1977 and FY 1978. Technical efforts were officially started when the annual operating plan was approved by DOE. A literature survey of existing coal-derived fuel properties was completed. Similarly, a literature survey of existing NO<sub>x</sub> emission models was made; a promising model was selected and modified for high fuel-bound nitrogen. Preliminary designs were completed for combustion model tests. A statistically designed experiment to study hot corrosion effects was initiated. One-hundred-hour accelerated burner rig tests are used to determine the effects of contaminant concentration and temperature on specimen weight change and thickness change. Potential fuel additives were evaluated in similar tests. Preliminary tests of a thermal barrier coating in heavily doped (accelerated) burner rig tests led to early coating spallation, confirming earlier pressurized passage results at Westinghouse. Fundamental coating optimization tests were started.

**PLANS FOR THE COMING YEAR** – The fuel characterization study will be completed. The NO<sub>x</sub> emission model will be modified and the results compared with experimental flame tube results. Flame tube hardware will be completed and testing will be started. Combustion concept hardware will also be completed. Promising fuel additives will be identified, and initial doped fuel screening tests will be completed in FY 1978. Several thermal barrier coating systems will be life tested to 1000 hours. Life testing with an actual coal-derived fuel (specified and supplied by DOE) will also be initiated. Preliminary airfoil cooling hole plugging tests will be completed. Finally, a study of thermal barrier coatings on liquid-cooled airfoils will be completed in FY 1978.

**DIAGNOSTICS ASSESSMENT FOR ADVANCED POWER SYSTEMS**

**SANDIA LABORATORIES**  
**DOE - \$250,000**  
**8/76 - Continuing**

**OBJECTIVES** – Diagnostic techniques will be assessed for application to characterizing the flow at the combustor exit and turbine inlet in advanced open cycle gas turbine systems. This assessment will include conventional as well as advanced (principally laser-based) techniques for the measure-

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\*FY 1977 funds provided for FY 1977 and FY 1978.



ment of properties of high-temperature, multiphase, and turbulent flows. The combination of close tolerances on specific turbine designs, hence knowledge of flow properties, and the potential flow variability arising from coal, coal-derived liquid and gaseous fuels, or heavy residual fuels emphasize the need to develop an adequate flow monitoring capability.

**RECENT WORK AND ACCOMPLISHMENTS** — An initial survey and evaluation have been completed of conventional and advanced diagnostic techniques for the determination of particulate size distribution, particulate mass loading density, gas velocity, gas temperature, and species concentrations. Those techniques which seem the most potentially useful for the advanced power systems were identified. Likely ranges of turbine inlet conditions expected in advanced systems were defined through discussions with turbine and combustor manufacturers, and electric utility representatives, and through consideration of previous experience at the Bureau of Mines and in the Australian Research Laboratories with direct pulverized coal-fired gas turbines. On the basis of these reviews, two regimes of advanced turbine operation were defined, combustor exhaust conditions were calculated, and the unique requirements imposed upon relevant diagnostic systems were evaluated. In general, it was concluded that a combination of advanced laser-based optical techniques and conventional probe sampling methods will be required to fulfill the diagnostic requirements of the advanced combustor/turbine systems.

**PLANS FOR THE COMING YEAR** — Sandia will continue to provide consultation and advice on diagnostic techniques pertinent to advanced power system projects in other laboratories, utilities, or industries. A comparative review of physical probing techniques for determination of the composition and mass loading densities of particulates entrained in the turbine inlet flow will be completed. Laboratory work will continue on four nonperturbing advanced laser-based techniques for measurement of gas temperature and composition, on the development of the two-color laser Doppler velocimeter, and on the development of laser-based techniques for in situ determination of particle size and number density. All techniques are designed to measure a time-averaged property of the flow field but with high spatial resolution. Crucial to the gas temperature and gas composition measurements will be the ability to discriminate against particulate-induced noise in the scattered laser light. Several techniques for effecting this discrimination will be evaluated in the laboratory.

## **FIRESIDE CORROSION**

**GENERAL ELECTRIC COMPANY, GAS TURBINE DIVISION**

**DOE - \$1,073,029**

**6/27/75 - 4/1/78**

**OBJECTIVES** — This program provides an engineering data base on the performance of selected current and near-term development gas turbine nozzle and bucket materials in gas environments expected from the combustion of coal-derived low-Btu gas and liquid fuels, at test temperatures up to 2200°F, and with nozzle and bucket internal cooling design practices typical of high-temperature operation incorporated in the tests. The program consists of three separate phases: initial tests, screening tests, and confirmation tests. The initial tests will define problems encountered when burning a coal-derived liquid fuel and low-Btu gas at pressure and velocity, at 1950°F. A turbine simulator at the General Electric R&D Center in Schenectady is being used to burn the coal-derived liquid fuel while another turbine simulator connected to the Morgantown Energy Research Center (MERC) gasifier has burned low-Btu gas. These data permit initial evaluations of corrosive effects

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and the likelihood of ash from the combustion of these fuels plugging cooling holes in aircooled nozzle vane and pin specimens. These data have been used in the design of a nozzle cascade for the confirmation tests. In the screening tests, 21 materials are being evaluated at 1600°F and 1800°F in small burner test stands using diesel oil doped with two ratios of Na and K; the results of these are utilized to estimate parts lives for various contaminant levels and compositions. The confirmation tests will be an evaluation of the best materials selected from the screening tests and state-of-the-art cooling design, at gas temperatures up to 2200°F, using the turbine simulator burning a coal-derived oil and low-Btu gas from the General Electric GEGAS D Gasifier.

**RECENT WORK AND ACCOMPLISHMENTS** — Initial tests using low-Btu gas were completed in July and August 1976 utilizing a turbine simulator with low-Btu gas provided by the fixed-bed gasifier and cleanup system at the DOE Morgantown Energy Research Center (MERC). The most significant results of these tests were that Na and K were detected at levels sufficient to initiate hot corrosion. Initial tests burning COED liquid fuel were conducted at GE. Again, species were found on hot gas path hardware which would have led to significant corrosion after several thousand hours of exposure at commercial firing temperatures. Further initial tests with a synthetic fuel to about 150 hours duration were completed with intentional ash and alkali added. Some cooling hole plugging occurred, but only very minor sulfidation at low ash levels. At high ash levels of 500 ppm, cooling hole plugging was unacceptable. Handling of liquid fuels has been examined with respect to corrosion of fuel system components. It was found that creosote must be handled in a narrow temperature range, low enough to avoid attack of pump components but high enough to avoid line plugging due to anthracene precipitation. Tests have been conducted to quantify fuel system corrosion rates. Samples of Fe, Cu, Zn, and alloys were exposed in creosote, H-coal liquids, and COED at 150°F for 8-25 hours. Cu was most affected (up to 49 mils/year), other materials much less.

Screening tests have been conducted in parallel with the initial tests to determine corrosive effects on 21 selected materials of alkali metals (Na, K) in combination. These include nickel and cobalt base alloys, directionally solidified eutectics, ceramics, and coating and cladding materials. Tests are in progress at 1600°F and 1800°F metal temperatures in small burner test stands, at 1 atm. Testing up to 8000 hours to date had included sodium levels from 79 to 116 ppm and potassium levels from 36 to 202 ppm. It has been observed that potassium, in combination with sodium, is more aggressive than sodium alone. Corrosion rates have generally monotonically increased with increasing mole percent potassium in deposits containing both sodium and potassium, over a range from pure Na<sub>2</sub>SO<sub>4</sub> to 23 mole percent K<sub>2</sub>SO<sub>4</sub>. However, the relative ranking of materials with regard to corrosion resistance remains unchanged by the addition of potassium. Alkali conversion studies have been initiated to determine whether or not alkali metals in minerals (silicates, aluminosilicates, etc.) convert to the corrosive soluble forms during combustion. An aromatic type petroleum fuel is doped with H-coal sludge in which alkali is present in water soluble and insoluble forms similar to coal. A water-cooled ash collection probe is inserted into the small burner test section to collect the deposits from this combusted mixture, whereupon the alkali metals are examined for their solubilities. The results of this testing are that (1) large amounts of Na, perhaps approaching 100 percent are "unlocked" in the combustion process to form water-soluble deposits, (2) only small amounts of K are "unlocked" in the combustion process to form water-soluble deposits, and (3) soluble deposits were found to be mixtures of Na<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>SO<sub>4</sub>.

be somewhat more corrosive to the austenitic materials. No significant differences were noted when the 800 series or cobalt based alloys were exposed while burning the various coals. The wastage was slight on most samples, although increasing with temperature and some enrichment of specific elements was observed in the deposits adjacent to the tube surface. Chromium, nickel, cobalt and tungsten all showed measurable increases.

Most of the coatings have suffered some loss in integrity from fabrication irregularities and/or porosity, particularly at higher temperatures. Fabrication defects in the In 617 alloy used in laboratory testing proved to be the starting point of grain boundary attack. The 617 coupons examined after field exposure seem to be free from this problem; thus more conclusive evaluation of the usefulness of this material is possible. The 671 clad does not appear to be providing the anticipated corrosion resistance at higher temperatures in either field or laboratory studies. In those samples containing butt welds between In 671 and In 800, the clad has been attacked preferentially over the In 800 or the weld metal. On the basis of preliminary field examination, the 12R72 and 316 alloys suffered the most significant wastage and do not appear to be satisfactory for extended service above 1200°F. Most of the coatings on the In 800 failed at least partially. Samples fabricated of 310, 800, and 188 have shown only superficial wastage as have the In 82 welds used between dissimilar metals.

**PLANS FOR THE COMING YEAR** – Chemical, physical, and metallographic examination of field probes, currently underway, will be completed and reported.

## PROCESSING AND UTILIZATION OF FOSSIL FUELS

NATIONAL ACADEMY OF SCIENCES

DOE - \$262,500

8/1/74-2/28/78

**OBJECTIVES** – A comprehensive evaluation of the status, technology, and research and development priorities will be derived for the major components of the coal processing and utilization system (i.e., coal mining, direct combustion, liquefaction, low-Btu gasification, and advanced power cycles). The objectives of the study are to provide: (1) an assessment of the priorities to be assigned to the components of a process, among processes within a given area, and among areas; (2) an outline of research and development needs, supportive research requirements (with particular emphasis on the role of universities), projected time schedules, and development strategies; (3) an assessment of environmental impact and technology requirements; and (4) an assessment of the impact of materials, capital, and manpower requirements. Foreign and domestic extraction and processing methods available or under development are considered, as are questions related to concepts, alternatives, engineering adequacy, costs, efficiency, applicability, process maturity, and possible rates of installation.

**RECENT WORK AND ACCOMPLISHMENTS** – Four reports have been published; two additional reports are in preparation. Upon publication of these two reports, *Assessment of Advanced Power Cycles Technology* and *Coal Mining*, the project will be completed.

**PLANS FOR THE COMING YEAR** – Contract work will be completed early in FY 1978.

## **FIRESIDE CORROSION: BOILER TUBES FOR ADVANCED POWER CYCLES**

**COMBUSTION ENGINEERING, INC.**  
**DOE - \$1,017,331**  
**6/30/75 - Continuing**

**OBJECTIVES** – The program is a combined laboratory and field investigation for testing, identification, and evaluation of commercial and advanced experimental materials and coatings exposed on controlled temperature probes under design conditions simulating advanced fuel power cycle combinations. Preliminary evaluation of selected materials and coatings was carried out in the KDL solid fuel fired test furnace using selected coal feedstocks of economic significance. The optimized materials are being evaluated in operating boilers using the same feedstocks for extended periods of time.

**RECENT WORK AND ACCOMPLISHMENTS** – Feedstock selection was made on the basis of long-range economics and geographical location of coal type, according to the contract. Exposure of both laboratory and field probes was made during combustion of the coal feedstocks utilized in the following: (1) Keystone Utilities Group at Keystone Station, Plum Creek Township, Pennsylvania; (2) Pacific Power and Light at Dave Johnston Station, Glenrock, Wyoming; (3) Industrial Generating Co. at Big Brown Station, Fairfield, Texas; and (4) Illinois Power Co. at Baldwin Station, Baldwin, Illinois. Work on this contract began July 7, 1975. Initial selection of alloys and coatings included known materials whose predicted 100,000-hour creep strength suggests possible service at the expected metal temperatures (1300-1700°F) of advanced power cycles. Selected materials were Tp 316, Sandvik 12R72, In 800H, In 802, In 617, Haynes 188, TP 310, and In 671 clads. It was assumed that materials would not be exposed in temperature ranges where use would be impractical. All four 300-hour tests while firing 40 to 50 tons of the selected coal feedstocks in the laboratory test facility have been completed. Probes containing the selected materials were exposed in the water-cooled “superheater” section. Field exposure of all probes for both 4000- and 8000-hour test periods has been completed. Probes specimens underwent chemical, physical, and metallurgical evaluation, which did not reveal accelerated wastage of the type observed on conventional superheaters operating in a metal temperature range of 1000-1300°F. Such wastage is caused by molten phase complex sulfates which are unstable at higher temperatures (3100°F). Although there was only slight wastage in all tests, the Dave Johnston and Baldwin coals appear to

## TECHNICAL INFORMATION SERVICE, IEA COAL RESEARCH

### NATIONAL COAL BOARD

DOE - \$150,177

11/20/75 - 3/31/83

**OBJECTIVES** – Information related to all aspects of coal technology is disseminated by the Technical Information Service so as to give guidance to workers in the field and to increase general awareness of the potentialities of coal and the coal industry in meeting future energy needs. The Service concentrates on the available information published in technical journals, conference papers, and research reports for current awareness or retrospective searching purposes. They fulfill this function through a computerized data base, a monthly current awareness journal *Coal Abstracts*, the production of reviews and bibliographies on subjects of interest to members, and the answering of public queries.

**RECENT WORK AND ACCOMPLISHMENTS** – To handle the data, a series of computer programs was required to process input and provide the necessary output. For the first stage, a simple off-line system will be used. An on-line version of this program is being developed so that interactive searching could be introduced at a later date. To ensure efficient retrieval, key words are used and a coal thesaurus is being developed. Establishment of the computerized data base provides both a source for retrospective searching and a means of disseminating the information through a computer type-set monthly abstract journal. A pre-production version of *Coal Abstracts* was produced in June 1977.

**PLANS FOR THE COMING YEAR** – Improvements to the input to the data base and *Coal Abstracts* will be a continuing task. Executive summaries and reviews will continue to be produced to a program agreed on by the Executive Committee. As the coal data base increases in size, the possible requirement for on-line access by organizations in member countries will arise. Preliminary consideration will be given to the preparation of a world directory of research in coal technology, and the supply of an SDI service to members.

## COAL-BASED FUEL DIESEL ENGINES FOR ELECTRIC POWER GENERATION

### THERMO ELECTRON CORPORATION

DOE - \$423,163

9/25/77 - 9/25/78

**OBJECTIVES** – This program will investigate the feasibility of operating the two stroke, slow speed, large bore diesel engine on coal based fuels for electric utility and industry cogeneration applications. The approach is to develop a referenced scientific and technological base from which the viability of the coal based fuel diesel can be assessed and from which a future development program can be structured. Emphasis will be on determining the most promising combustion characteristics and injection techniques for selected coal based fuels in this type of diesel engine. The two stroke, slow speed, large bore diesel engine offers the potential for efficient, economic generation of power using a variety of coal based fuels.

**RECENT WORK AND ACCOMPLISHMENTS** – This is a new program with all work to be performed in the coming year.

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**PLANS FOR THE COMING YEAR** – Key technical and economic characteristics of diesel engines using coal based fuels will be investigated by conducting engineering tests on an existing single cylinder research engine. The tests will be run with three fuels beginning with a relatively clean de-ashed and desulfurized COED fuel and progressively approaching the SRC type fuel. Fuel injection studies will investigate the most promising methods for handling and injecting the highly viscous SRC type fuels. Conceptual designs of overall power plant configurations will be established to provide a basis for economic analysis and evaluation of representative coal based fuel diesel systems. The results of the research engine tests, the fuel injection studies, and the economic analysis will be used to identify key component development areas and to formulate a detailed program plan for coal based fuel diesel power plant technology development.

## **CLOSED-CYCLE GAS TURBINE PRIMARY HEATER PROGRAM**

**ROCKWELL INTERNATIONAL CORPORATION**

**DOE - \$737,293**

**3/11/77 - 3/11/78**

**OBJECTIVES** – Coal-fueled combustion heat exchanger technology will be advanced for use with high-temperature closed cycle gas turbine/Rankine power conversion systems to a state of technology readiness, i.e., to a point where no major risks remain for full-scale commercial development. Two parallel approaches are being undertaken in order to assure that the desired goal is accomplished. The first approach will assume the direct combustion of coal in the combustion heat exchanger. The combustion heat exchanger will have a nominal working fluid turbine inlet temperature of 1550°F. The second approach will be directed toward the evaluation of combustion heat exchangers using turbine inlet temperatures in the range of 1750°F and higher, requiring the use of nonmetallic heat exchange surfaces. In addition, the gas turbine may require either blade cooling or the use of higher temperature blade materials than are possible for use in conventional gas turbines. In the event that direct coal firing of the primary heat exchanger proves infeasible, an alternative which may be considered is that of using coal-derived fuels, recognizing the cost and efficiency consequences of the coal conversion process. A critical component in the closed cycle gas turbine system is the primary heat source heat exchanger. This area of the power conversion system is the most critical to the successful application of the closed cycle gas turbine system. Increased fluid working temperature and high stresses are the reasons that the problem is more serious than with previous closed cycle applications operating in the coal combustion gas environment.

**RECENT WORK AND ACCOMPLISHMENTS** – Current Phase I contractual work will culminate in four heat exchanger designs, two at the 1550°F turbine inlet temperature condition, and two at the 1750°F or above condition. In addition, key features of these designs will be identified and an R&D program described which will allow the required technology to be reduced to practice.

**PLANS FOR THE COMING YEAR** – A task added to the contract will evaluate the Phase I heater designs as part of closed cycle gas turbine systems which could be used as cogeneration systems. Later in the year, Phase II of the main contract is expected to be initiated which will consist of conducting the necessary R&D work which can prove the designs proposed are feasible.

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- Ad Hoc Panel on Direct Combustion of Coal, Committee on Processing and Utilization of Fossil Fuels. *Assessment of Advanced Technology for Direct Combustion of Coal*. Washington, D.C.: National Academy of Sciences, 1977.
- Ad Hoc Panel on Liquefaction of Coal, Committee on Processing and Utilization of Fossil Fuels. *Assessment of Technology for the Liquefaction of Coal*. Washington, D.C.: National Academy of Sciences, 1977.
- Ad Hoc Panel on Liquefaction of Coal, Committee on Processing and Utilization of Fossil Fuels. *Assessment of Technology for the Liquefaction of Coal: Summary*. Washington, D.C. National Academy of Sciences, 1977.
- Ad Hoc Panel on Low-Btu Gasification of Coal, Committee on Processing and Utilization of Fossil Fuels. *Assessment of Low- and Intermediate-Btu Gasification of Coal*, Washington, D.C.: National Academy of Sciences, 1977.
- Hurn, R.W. "Performance Characteristics of a Motor Gasoline Produced From Oil Shale." *Proceedings of the Ninth Oil Shale Symposium*, Oct. 1976. Colorado School of Mines Quarterly, 71, No. 4: 33-38 (1976).
- . "Characteristics of Conventional Fuels From Non-Petroleum Sources—An Experimental Study." Presented at the General Motors Fourth International Symposium, Oct. 1976. *Future Automotive Fuels—Prospects, Performance, Perspective*. New York: Plenum Press, 1977.
- Louis, J.F., and Topping, R.F. *Combined Cycle Research Program, Quarterly Technical Progress Report No. 1*. Massachusetts Institute of Technology Energy Laboratory, Oct. 1976.
- . *Report No. 2*, Jan. 1977.
- . *Report No. 3*, Apr. 1977.
- . *First Annual Report*, July 1977.

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# ***DIRECT COMBUSTION***

Coal is the Nation's most abundant domestic fuel. Based on proven resources recoverable with current technology, energy available from coal is nearly 30 times greater than that available from oil and natural gas combined. Yet, because of the former low price of oil and gas, coal represents less than 55 percent of the fossil fuel consumed by steam-electric utilities. National emission standards for stationary coal-fired steam generators limit sulfur dioxide ( $\text{SO}_2$ ) emissions to 1.2 lb/MM Btu, and nitrogen oxides ( $\text{NO}_x$ ) to 0.7 lb/MM Btu. Sulfur dioxide is present only in relatively low concentrations, and the high cost of removing it from large volumes of stack gases has restricted the use of sulfur coals. Approximately 50 percent of coal presently being mined can meet emission standards for new sources without some kind of  $\text{SO}_2$  emission suppression. Because fluidized-bed combustion (FBC) boilers (atmospheric and pressurized) should produce considerably less  $\text{SO}_x$  and  $\text{NO}_x$  than conventional coal-fired boilers, the technology should enable increased coal utilization in an environmentally acceptable manner. Particulate matter emissions can also be reduced to environmentally acceptable levels, but greater quantities of fine particulates appear to be emitted from FBCs. The major environmental concern with FBCs is, however, disposal of the large quantities of solid wastes produced from the fly ash and the bed itself. Many programs are presently underway to study the characteristics and possible uses for the materials.

The objectives of the Direct Combustion program are to develop fluidized-bed combustion systems capable of directly burning high-sulfur coals of all ranks and quality in an environmentally acceptable manner, to develop technology to substitute coal for a substantial portion of oil in combustors capable of firing coal-oil mixtures, and to improve reliability and efficiency of present boilers. To enable coal resources to be fully exploited, research efforts are being directed toward development of environmentally acceptable methods of combustion and improved combustion efficiency. To achieve this goal, the total effort has been integrated in terms of both broad technical approach and specific technical components. Specific areas of research and development include:

- Fluidized-bed combustion (atmospheric and pressurized) with particular attention to achieving high-combustion efficiency, acceptable component durability, minimum emission of particulates and sulfur and nitrogen oxides, and reliable operation of combined-cycle systems
- Combustion and heat transfer characteristics of chars, coal-oil slurries, solvent-refined coal, and coal-derived liquid fuels when burned in conventional furnaces, and the application of such data to improved combustor design
- Causes of adherent slag and ash deposits, and development of methods for minimizing these efficiency-degrading problems
- Identification and control of toxic elements evolved during the direct combustion of coal.



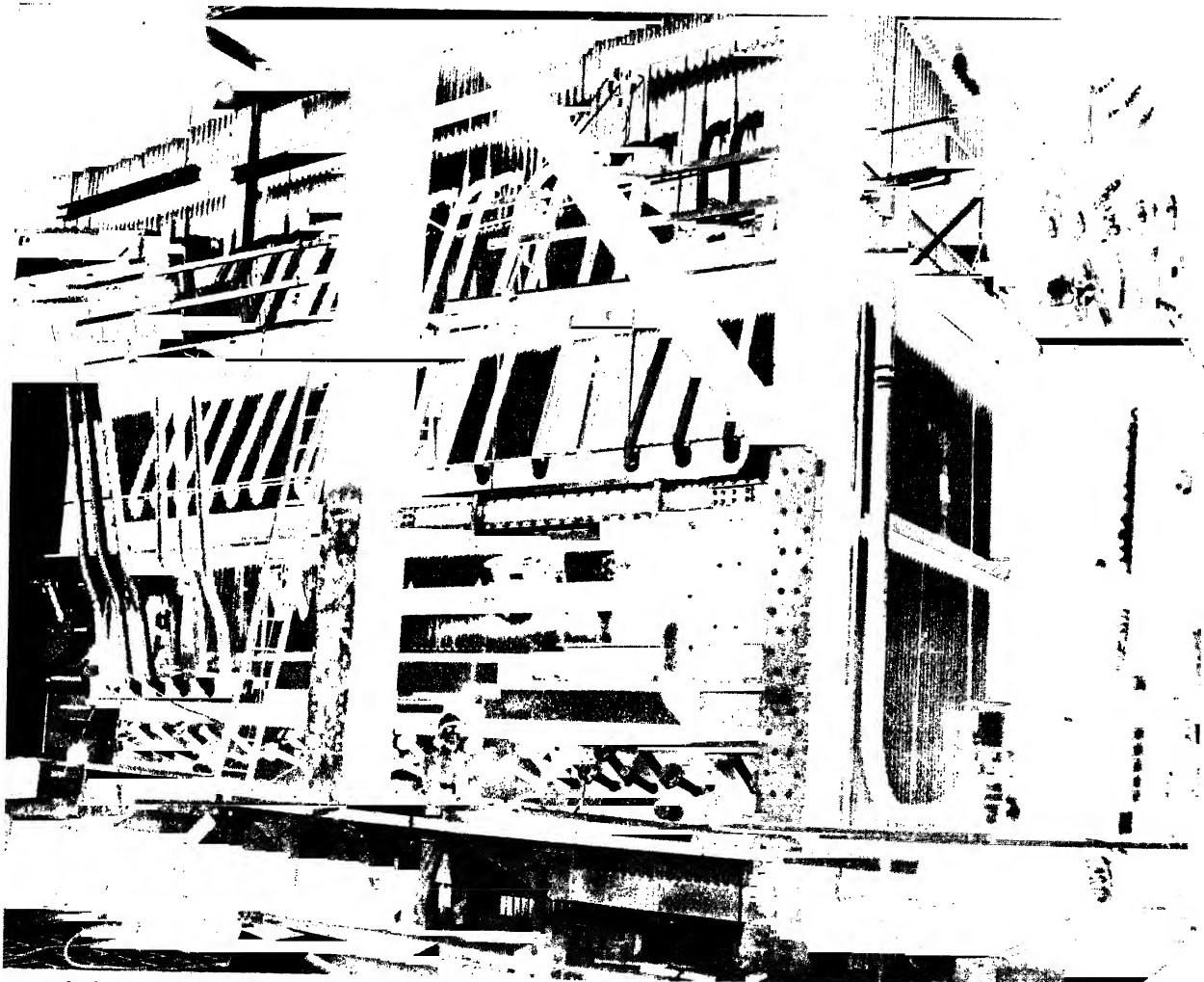
## INDUSTRIAL APPLICATION OF FLUIDIZED-BED COMBUSTION

COMBUSTION ENGINEERING INCORPORATED

DOE - \$5,880,402; CEI - \$1,810,938

6/76 - 3/81

**OBJECTIVES** — This task is to develop a commercial atmospheric, natural-circulation, fluidized-bed steam generator designed as a shop-assembled unit and capable of shipment by rail. The boiler must be able to fire coal while meeting all environmental standards without the use of auxiliary sulfur removal equipment and be competitive with other current methods of coal firing for industrial practice. The work encompasses cold-flow modeling studies in a bench-scale fluidized-bed unit; design, construction, and testing of a subscale test unit; and design, construction and testing of a full-size 50,000 lb/hr steam industrial plant. The full-size demonstration plant will be located at the U.S. Naval Base, Great Lakes, Illinois, and will tie into existing power-generation and space-heating facilities. The fluidized-bed combustion concept has the greatest potential of all the available coal-firing systems to provide an economical, environmentally acceptable industrial boiler plant. Fluidized-bed combustion (FBC) has been under development for some time, and the principal need today is to successfully demonstrate this process for the direct combustion of coal.



*Industrial-Size 30-Mw "Clean Energy" Fluidized-Bed Coal Combustion Boiler in Successful Operation at Rivesville, West Virginia; Innovative Process Is Capable of Eliminating 95 Percent of Sulfur Dioxide Emissions.*

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**RECENT WORK AND ACCOMPLISHMENTS** — The cold-flow testing of the two-dimensional and three-dimensional fluidized-bed module was completed except for some testing of bubble growth and slugging. Limited cold-flow modeling to study defluidization of sections of the bed as a method of load turndown and control was completed. The design, material procurement, and initial construction of the subscale fluidized-bed unit was also completed. Materials are on site and most of the major equipment has been installed. Preliminary design of the demonstration plant has begun and includes: preparation of plant layout drawings, development of a boiler startup procedure (based on transfer of ignition energy from an operating bed section to an adjacent fluidized section), development of an air inlet plenum chamber with independent air-flow measurement and control, development of a fuel handling system, and preparation of system design criteria and equipment specifications. The termination points for all plant systems that connect to existing Navy facilities were also identified.

**PLANS FOR THE COMING YEAR** — The cold flow modeling effort will be completed. Subscale unit construction, shakedown operation, and testing will be completed. The results of the test program will be used in the design of the demonstration plant. Final design of the demonstration plant systems and selection of equipment will occur during the next year. Plant construction is also scheduled to start under Phase II of the program.

## INDUSTRIAL APPLICATION OF FLUIDIZED-BED COMBUSTION

EXXON RESEARCH AND ENGINEERING COMPANY

DOE - \$3,274,757; Exxon - \$733,618

6/30/76 - 3/81

**OBJECTIVES** — This program is to evaluate the potential application of fluidized-bed combustion (FBC) technology to indirect-fired process heaters. If early laboratory and engineering studies indicate the process to be technically and economically viable, a secondary objective will be to demonstrate the application of a coal-fired fluidized-bed heater in a petroleum refinery environment. The program strategy is to build on available boiler-oriented FBC technology. Areas common to both steam-generating boilers and process heaters will not be intentionally advanced by this program; however, the results of complementary programs in the boiler area will be considered in the assessment of potential heater applications.

**RECENT WORK AND ACCOMPLISHMENTS** — Two pertinent areas being investigated concern the effects of FBC unit performance on larger diameter tubes and on hydrocarbon coking. Flow-modeling studies have been completed defining the optimum and range of acceptable tube-bundle configurations that could be used in commercial heater designs using tubes of 2-inch- to 6-inch-diameter sizes. For the first time, extensive conductive/convective heat transfer data have been obtained on 4-inch- and 6-inch-diameter tubes (both single tubes and bundles) immersed in a fluidized bed. These data include both overall average heat transfer rates and peripheral maldistribution patterns as a function of tube location, surface orientation, and fluidization velocity. A second laboratory facility has been constructed and is now in startup that will evaluate internal tube coking in hydrocarbon service as a function of heat flux rate, mass flow, bulk fluid temperature, and inside film temperature. The data from these tests will help determine to what extent future heater designs can take advantage of the high heat flux rates available in a fluidized bed while still maintaining a reasonable rate of tube coking and unit service factor.

**PLANS FOR THE COMING YEAR** – The hydrocarbon tube coking tests will be completed. A 16 MMBtu/hr coal-fired fluidized-bed test unit will be built and put into service to begin generating data on overall heat transfer coefficients and to demonstrate operability and control of a coal-fired FBC unit.

#### INDUSTRIAL APPLICATION OF FLUIDIZED-BED COMBUSTION

FLUIDYNE ENGINEERING CORPORATION  
DOE - \$2,878,385; Fluidyne - \$1,537,227  
5/12/77 - 9/1/81

**OBJECTIVES** – This program involves the design, construction, and operation of an atmospheric fluidized-bed air heater burning high-sulfur coal in an environmentally acceptable manner with the load-following requirements encountered in an industrial plant. The plant site is the Owatonna Tool Company of Owatonna, Minnesota. Operation over a period of 3 years is planned, during which data will be obtained that will provide a basis for design and construction of commercial units.

**RECENT WORK AND ACCOMPLISHMENTS** – The program is divided into two phases: technology development and design, and construction and operation. Work thus far has included development tests and system design. Development tests with an 18-inch-square bed have been run with Iowa coal, Tennessee coal, and petroleum coke. Results have shown that the combustion efficiency, sulfur retention (using locally available dolomite), and in-bed heat exchanger design are all satisfactory. These results were applied to the design of a larger test unit, having a bed size of 40-by-64 inches. This unit is essentially a vertical slice of one of the full-sized combustors. Equipment operation and design concept confirmation tests began in this unit in April 1977. Particular attention has been given to optimizing performance of the automatic ignition and control systems, and to the response to load changes and upset conditions. A 500-hr test was conducted with the 40-by-64-inch combustor to demonstrate equipment performance over a long period of continuous operation. This test was completed and included both long-term fixed-load and variable-load operation.

**PLANS FOR THE COMING YEAR** – The project schedule calls for the demonstration plant to be in full operation for the 1979-1980 heating season, during which operating and maintenance data will be obtained.

#### INDUSTRIAL APPLICATION OF FLUIDIZED-BED COMBUSTION

GEORGETOWN UNIVERSITY  
DOE - \$9,920,696; Georgetown University - \$4,611,962  
6/30/76-4/82

**OBJECTIVES** – The program includes the design, construction, testing, and operation of a 100,000 lb/hr fluidized-bed, coal-fired saturated steam (625 psig), demonstration boiler plant at Georgetown University, Washington, D.C. The plant will be fueled with low-quality high-sulfur content (3.5 to 4 percent) coal. It is intended to prove that this type of plant is adaptable to central heating-cooling plants for existing and future health and educational facilities. The plant will be designed to be aesthetically and operationally acceptable in a university/medical complex environment; built and operated with time schedules and costs equal to or better than conventional

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coal-fired plants; operated reliably and efficiently by personnel with experience typical of institutional boiler plants; capable of operating efficiently over wide swings in load with and without oil/gas-fired boilers in parallel; and capable of meeting air pollution standards.

**RECENT WORK AND ACCOMPLISHMENTS** — Design of the plant and boiler was completed at the end of September 1977. Significant design changes were investigated by the Contractor and approved by DOE: increasing the operating pressure of the boiler to 625 psig, revising the feed system to provide stoker feed, and revising the byproduct removal system to separate the spent bed material from the fly ash. Construction bid packages, including drawings and specifications, were completed for general construction, steel fabrication and erection, mechanical and electrical work. The general construction and steel fabrication and erection contracts were awarded in late September and award of the mechanical and electrical work is scheduled for October. Procurement of prepurchased equipment was started, and purchase orders were issued for several of the long-lead items. The G.U. emissions monitoring plan was approved by DOE and EPA, and procurement of the necessary equipment was initiated.

**PLANS FOR THE COMING YEAR** — Construction of the plant will begin in October 1977. The remaining construction contracts, covering electrical, mechanical and instrumentation work, will be awarded at that time. Boiler fabrication will be completed and field erection of the boiler will be started in early 1978. Remaining items of prepurchased equipment will be procured, and all items will be installed during this period. Procurement of coal and limestone will be initiated and contracts signed. Development of operating manuals and startup procedures will begin. Equipment for the emission monitoring program will be set up and calibrated, and detailed procedures developed. This plant is scheduled for completion during the first calendar quarter of 1979, and startup testing will begin immediately thereafter.

## INDUSTRIAL APPLICATION OF FLUIDIZED-BED COMBUSTION

BATTELLE, COLUMBUS LABORATORIES  
DOE - \$5,256,355  
6/76 - 4/83

**OBJECTIVES** — This program is to investigate the fluidized-bed combustion process at substantially higher bed velocities (30 to 40 ft/sec) than the conventional range (6 to 12 ft/sec). The claims of higher heat release rates, higher overall heat transfer coefficients, and improved Ca/S mol ratio capture are based on earlier work performed in a 6-in.-diameter combustor. The contract will begin with the design, construction, and startup of a subscale experimental unit having a bed cross section of about 2.5 sq ft. Operation of this unit will provide data and direction for the detailed configuration and design of a boiler to produce 25,000-lb/hr 100-psig steam. Both subscale and boiler systems will be located at Battelle, Columbus Laboratories, Columbus, Ohio.

**RECENT WORK AND ACCOMPLISHMENTS** — The subscale experimental unit was designed, constructed, started, and operated during the reporting period. Ranges of fuel and sorbent feed rates were evaluated. Sorbent and fuel particle-size distributions were investigated relative to Ca/S mol ratio captures, combustion efficiencies, and system temperature distributions. Materials of construction were closely watched because of the high rates of particle movement in the system. Sufficient data were not obtained to definitize the relationship, but it became evident that the 2.5-sq-ft combustor exhibited differences in operating data when compared to the 6-in.-diameter unit. The extent of those differences has yet to be quantified.

**PLANS FOR THE COMING YEAR** – Work on the subscale experimental unit will continue into the year and be completed prior to year end. Definition of all operating parameters will be sufficiently well along to design the demonstration system. These data will first provide the necessary guidance on proceeding to the demonstration phase of the program. If there are indications that this fast bed approach can provide process improvements, enhanced operation, and/or beneficial economics, it will be evaluated as a prototype boiler. System construction will then follow long-delivery equipment procurement and detailed design.

## **INDUSTRIAL APPLICATION OF FLUIDIZED-BED COMBUSTION**

**ARTHUR G. MCKEE & COMPANY**  
DOE - \$1,021,000  
6/30/76 - 6/30/78

**OBJECTIVES** – This task will provide technical assistance services to DOE for developing and demonstrating the industrial applications of fluidized-bed combustion. These efforts include monitoring each of the five participating contractors (Battelle, CEI, Exxon, Fluidyne, and Georgetown U.) for the various program planning and scheduling functions; providing technical assistance on design, both preliminary and detailed; monitoring construction activities by the contractors including coordination of design, scheduling activities, and manpower and cost control; reviewing contractor test programs related to testing of components, subsystems, instrumentation, controls, and data acquisition; providing assistance in the implementation of test programs designed for the operation and maintenance in both the demonstration and pre-demo modes; providing assistance on data management involving scaleup to demonstration and commercial systems; collecting and evaluating all operational data related to reliability of components, subsystems, and the composite facility; and assimilating and evaluating all data related to capital and operating costs of the contracted application compared to alternative systems.

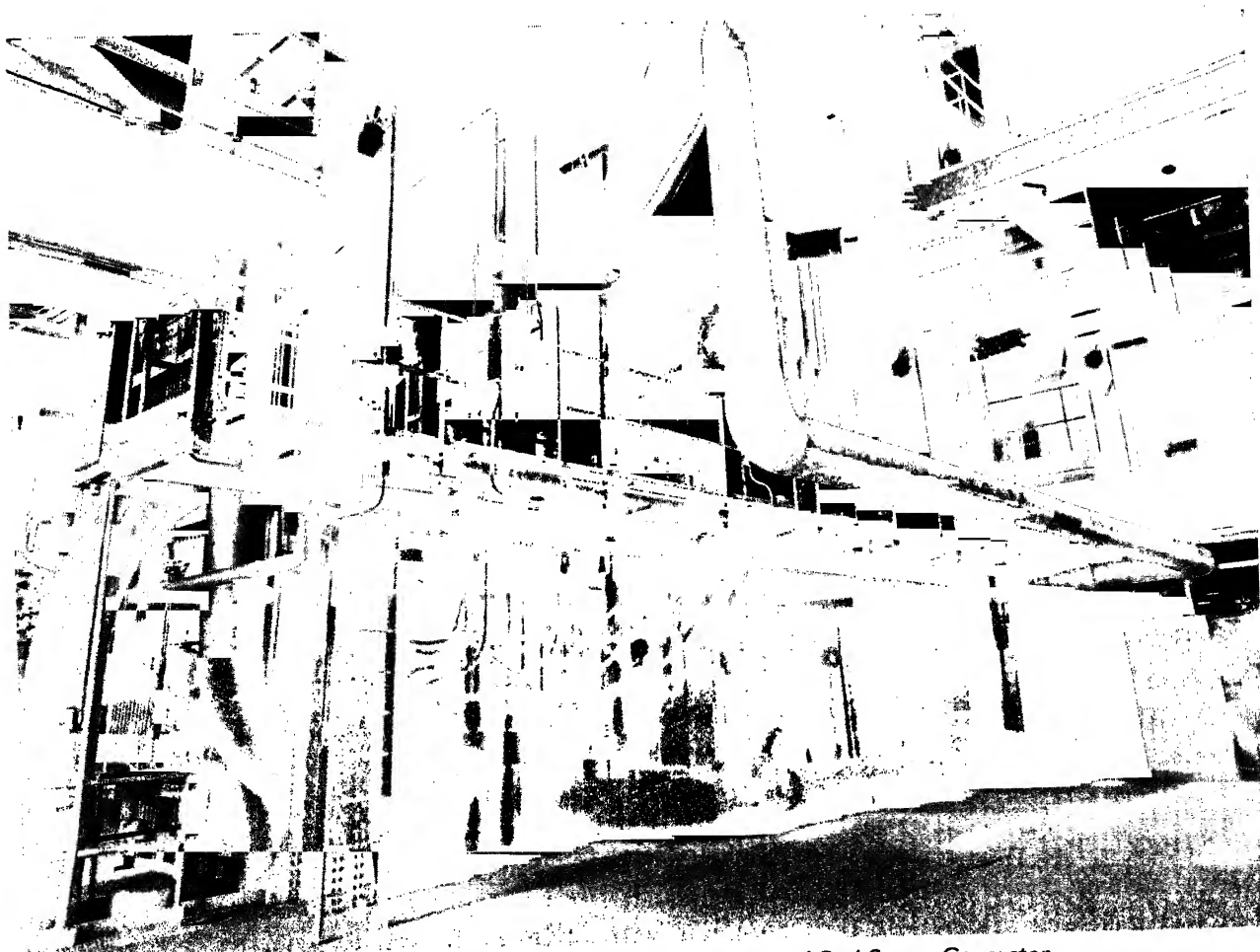
**RECENT WORK AND ACCOMPLISHMENTS** – Work and test plans, schedules, preliminary drawings, and equipment specifications were reviewed. Technical reviews were also held with the various contractors. For those contractors who were involved in subscale testwork, 30 percent design reviews were held to discuss and comment on the progress to date for design and construction of the subscale units. Several technical review meetings were also held with a contractor who is involved in the demonstration phase. These demonstration plant design reviews were conducted at the 30, 50, and 80 percent points of final design and subsequently reviews of each of five separate subcontract bid packages, as well as bid tabulations, were completed prior to contract award. Technical comments and recommendations on both the subscale and demonstration units were presented to DOE in the areas of plant layout, electrical, mechanical, instrumentation, and process. Contractor monthly and quarterly technical reports, as well as financial reports, were reviewed in detail and comments were submitted to DOE. Presently, one participant is in the construction phase

Construction monitoring has begun for one demonstration plant which is now under construction and scheduled for completion during the first quarter of 1979. Strong emphasis will be placed on cost and schedule control to assure timely completion within allocated budgets.

## UTILITY APPLICATIONS, MULTICELL FLUIDIZED-BED BOILER

POPE, EVANS AND ROBBINS INC.  
DOE - \$28,586,799  
10/72 - 3/78

**OBJECTIVES** – This program involves designing, constructing, and operating prototype multicell fluidized-bed boiler (MFB) at an electric utility power station as a method of burning high-sulfur coals in an environmentally acceptable manner and without excessive maintenance problems. The system is to have a capacity of 30 Mw<sub>e</sub> and is to be operated under typical electric utility conditions. In addition, PER is conducting a laboratory research program to optimize certain boiler and operational features. Experience and information gained from these programs will be used for scaling up to larger MFB systems. Advantages of the MFB are that sulfur dioxide and nitrogen oxide emissions from the combustion of high sulfur coals are maintained well within EPA standards. Also, MFB is a cost-effective alternative to fuel gas desulfurization systems, the sulfur dioxide being captured during combustion by the use of limestone as the sorbent in the fluidized bed.



*30 Mw<sub>e</sub> (300,000 lb, 15 t/hr) Capacity Multicell Fluidized-Bed Steam Generator*

**RECENT WORK AND ACCOMPLISHMENTS** – Field erection of the MFB boiler in the Monongahela Power Company's Rivesville, W. Va. plant was completed during 1976. Construction of the MFB plant was completed and accepted in March 1977, after the equipment and systems were operated in the cold and hot mode for checkout and calibration. First successful coal firing of the boiler was accomplished on December 7, 1976. Intermittent 2-, 3-, and 4-cell operation was accomplished during the Spring and Summer of 1977. To improve potential for long-term continuous operation of the coal feed system, coal purchases were limited to dry, screened coal in the  $-\frac{1}{2}$  to  $+\frac{1}{4}$  in size range. In addition, repairs and modifications were made to the ignitor, feedwater, fuel receiving, and fuel and limestone feed systems. Installation of additional supporting instrumentation has progressed to the stage where all control loops, except bed height, have now been calibrated and are operable in the automatic mode. On August 26, 1977, the MFB plant was formally dedicated by Senator Robert C. Byrd. First commercial operation of the boiler was accomplished on September 16, 1977, when steam produced in the MFB was used for generation of electricity by Monongahela Power Company's Turbogenerator No. 5 for 1.7 hours at a 6 Mw<sub>e</sub> level. On September 30, 1977, the MFB was operated successfully at its full design capacity of 300,000 lb/hr. A variety of test programs were conducted in the Alexandria, Virginia, FBC laboratory, utilizing new systems installed for carbon burnup cell operation and automatic combustion control. Tests in the carbon burnup cell mode indicated higher overall combustion efficiency than those previously experienced in small combustors. Data were also developed on the effect of injecting coal into the bed in different manners and locations. Fly-ash recycle tests were conducted to verify system performance and to provide data on fly-ash characteristics for dust collection system designs. Automatic control and transient response tests were performed to verify systems design and operability, and to provide data for dynamic modeling. Alternative lightoff techniques were explored and coal feeding educators tested to provide operating and design information needed for the multicell FBC boiler test program. In a continuous program to improve sorbent utilization, agglomerates of limestone were tested.

**PLANS FOR THE COMING YEAR** – The Alexandria combustion laboratory will complete the design and installation of a new steam generator (PDU) with submerged heat transfer surfaces in the 3-ft-by-3-ft cross-section unit, to accelerate optimization of combustion efficiency and sulfur capture, while providing a facility for continued hardware development work. The Rivesville MFB Plant is being upgraded by the installation of new equipment in the coal handling and feeding systems; following this 3-month upgrading period, continuous-mode plant operation will be carried out, during which time detailed heat and material balances will be prepared for various boiler load conditions. Commercial feasibility demonstration operation for steady-state heat and material balances, transient response, erosion, corrosion, and environmental control evaluation will also be performed.

## CONCEPTUAL DESIGN OF ELECTRIC POWER GENERATING PLANT

STONE & WEBSTER ENGINEERING CORPORATION

DOE - \$1,354,000

1/19/77 - 1/31/78

**OBJECTIVES** – This project aims to define the technical feasibility and economics associated with a 570-Mw electric power generating plant equipped with an atmospheric fluid bed (AFB) boiler. The project has been undertaken in conjunction with Pope, Evans and Robbins to perform the following tasks: (1) define an ongoing pulverized coal-fired base plant equipped with a wet



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limestone FGD, (2) have boiler manufacturers, Foster Wheeler Energy Corporation (FW) and The Babcock & Wilcox Company (B&W), design an AFB boiler and ancillary equipment to replace the base plant boiler and ancillary equipment, (3) incorporate the AFB boiler into the total plant, (4) develop the capital and operating costs of the AFB plant, and (5) evaluate the economics and technical feasibility of AFB designs to each other and to the base plant. The project results will permit an evaluation of the technical and economic merits of an AFB facility to ascertain whether, in a full-scale size, the AFB technology remains a viable solution to our dependence on oil and gas. In addition, by concentrating on the larger scale, alternate systems and equipment to those currently in use at Alexandria, Rivesville, and other AFB laboratory facilities will be conceived and/or extrapolations of existing equipment/systems design bases, etc., will identify areas which require additional development and/or testing.

**RECENT WORK AND ACCOMPLISHMENTS** – The conceptual design and capital cost estimate of the 570-Mw AFB boilers have been completed by both B&W and FW. These equipment designs have been incorporated into essentially duplicate total electric power generating plants. Plant conceptual design drawings and plant capital and operating costs have been developed. A computer model of the FW once-through AFB design's response to load changes is being developed and is expected to be completed by January 31, 1978. The evaluation of the capital and operating costs of the AFB design is underway and is expected to be completed by January 31, 1978.

**PLANS FOR THE COMING YEAR** – It is anticipated that the activities associated with the existing scope of work for this contract will be completed by January 31, 1978.

### **AFBC UTILITY STEAM GENERATOR**

**TENNESSEE VALLEY AUTHORITY**

**DOE - \$1,250,000**

**1/77 - 5/78**

**OBJECTIVES** – Preliminary designs will be developed and evaluated for a demonstration size utility fluidized-bed boiler system which will be capable of using high-sulfur eastern coal to generate electric power in an economically and environmentally acceptable manner. A power plant of approximately 200 Mw using Western Kentucky coal containing 4.5 percent sulfur and 15.5 percent ash was selected for the study. Supporting activities include the development of capital and operating costs, the definition of areas where additional research and development is required, an evaluation of potential commercial applicability, and the coordination and integration of activities related to the AFBC demo plant development. The atmospheric fluidized bed combustion process has been proposed as an attractive alternative to a conventional pulverized coal-fired boiler with flue gas desulfurization. The AFBC boiler offers the advantage of SO<sub>2</sub> removal within the limestone bed, reduced NO<sub>x</sub> emissions resulting from low combustion temperatures, high heat transfer rates, and high combustion release rates. These advantages serve as the impetus for developing the AFBC steam generator for utility and industrial applications as well. A substantial amount of data exists from small-scale AFBC facilities used in obtaining basic research information. The gap between these facilities and commercial reality will be significantly narrowed in the coming months as data and operating experience are gained from the Department of Energy's Rivesville, West Virginia, pilot plant and from the Electric Power Research Institute's (EPRI) pilot plant at Alliance, Ohio. A demonstration plant on the order of 200-Mw size is considered an essential last step prior to commercialization in order to provide economic data and full-scale simulations necessary to



guarantee commercial success such as startup and shutdown procedures, load following, turn down control, sulfur capture,  $\text{NO}_x$  and particulate control, and combustion efficiency.

**RECENT WORK AND ACCOMPLISHMENTS** – Contracts for the preliminary designs of the AFBC steam generator and auxiliaries were signed and work began in January 1977 by Babcock and Wilcox (B&W) and Combustion Engineering (CE) and in February 1977 by Fluidized Combustion Company (FCC). All three designs exhibit one major innovation compared with previous utility AFBC designs in the United States and the United Kingdom. This consists of a single large overhead chamber, similar to a furnace of a conventional boiler, followed by a convection pass of conventional design. Another innovative feature is CE's ranch style bed arrangement which places all bed modules at the same elevation to facilitate support and access. Two contractors have selected a sub-critical recirculating steam system. Main bed areas to achieve 200 Mw vary from 2000 to 5600  $\text{ft}^2$  due to a wide selection of superficial velocities (4-12 ft/sec). This selection also has a strong impact on the range of predicted Ca/S which is presently 2.3 to 4.0 for the three designs.

**PLANS FOR THE COMING YEAR** – CE and FW final reports are due to TVA by December 31, 1977, and the B&W final report is due February 11, 1978. A TVA final report evaluating the contractor work is due to DOE in draft form March 10, 1978, and in final form May 26, 1978.

#### AFBC ELECTRIC POWER GENERATING PLANT

BURNS AND ROE, INC.  
DOE - \$1,320,972  
1/10/77 - 1/10/78

**OBJECTIVES** – A conceptual design will be prepared for an electric power generating plant that will directly combust noncompliance western coal in an atmospheric fluidized-bed boiler and generate electric power in an environmentally acceptable manner. The program includes the following specific objectives: (1) to design a technically feasible, reliable, and efficient combination of unit processes and equipment that will directly combust noncompliance western coal and produce electric power in an environmentally acceptable manner, and (2) to project the capital, operating, and product costs for a central station electric power generating plant.

**RECENT WORK AND ACCOMPLISHMENTS** – Two tasks, initial plant criteria and design alternatives, were completed and a report was issued to DOE. This report served as the basis for engineering decisionmaking during the conceptual design of the AFBC electric power plant. The design alternatives to be analyzed included plant size, modular boiler size, steam conditions, Ca/S mole ratio, and sorbent regeneration. Under the third task, fuel preparation, a report on "Coal Supply Systems" was issued to DOE in May 1977, presenting the rationale for selecting a design coal for this program, the basis for the coal cost, and the description, specification and cost for the equipment selected for receiving, unloading, crushing, drying, sizing, handling, storing, and feeding the coal for a nominal 600  $\text{Mw}_e$  AFBC power plant. The design coal is a noncompliance subbituminous coal from the Felix coal bed in the Powder River Basin in Wyoming. It has a design sulfur content of 0.89 percent and a heating value of 8053 Btu/lb, and thus required 46 percent  $\text{SO}_2$  removal to meet the emission standard of 1.2 lbs  $\text{SO}_2/10^6$  Btu fired. Under the fourth task, Burns and Roe issued a specification for the engineering and conceptual design of an AFBC steam generator and accessories, and subsequently awarded a subcontract to Combustion Engineering (CE). A report describing CE's AFBC boiler design was submitted to DOE in November 1977. A

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report on "Sorbent Systems" was issued to DOE in July 1977 in accordance with the fifth task. This report included system descriptions, equipment specifications and costs for sorbent preparation, handling, storage and disposal, as well as the evaluation of a once-through versus a regenerative sorbent system. On the basis of this evaluation, a once-through sorbent system utilizing limestone with a 90 percent minimum  $\text{CaCO}_3$  purity was selected for this program.

Under the sixth task, definition of major equipment and system design parameters, a report on "System Design Analysis" was issued to DOE in July 1977. This report included the analyses of plant size, steam conditions, reheat cycles, feedwater heaters, cooling towers, and condenser/cooling tower optimization. Technical specifications were prepared under the seventh task, and subsequently issued to equipment suppliers to obtain budgetary estimates. The eighth task, design of the commercial plant, has been recently completed, incorporating the results of the previous tasks. Tasks 9 and 10, environmental and economic analyses, are currently in progress. Task 11, miscellaneous studies, includes a number of studies not specified in the original scope of work but which DOE and B&R agree should be addressed. These include (1) a more detailed analysis of control procedures involving start-up, shutdown, and off-design load operation; (2) a market survey to assess the potential impact of a proven AFBC technology, (3) a design and economic analysis of installing an AFBC unit as a hook-on to an existing gas or oil fired unit, (4) an analysis of design considerations and cost of operating a specific AFBC combustor with various coals, and (5) a sensitivity analysis on the increased cost of an AFBC power plant and of a conventional pulverized coal-fired plant with scrubbers due to more stringent emission standards. Task 12, the final report, is scheduled to be completed in January 1978.

**PLANS FOR THE COMING YEAR** – Tasks 9-12, as described above, will be completed. The final report is scheduled to be issued to DOE in January 1978. This report will present a description of the program, the AFBC plant design, and economic and environmental analyses of the AFBC electric power plant. Recommendations regarding AFBC demonstration plants and future AFBC R&D efforts will be presented.

#### **ATMOSPHERIC FBC/COMPONENT TEST AND INTEGRATION UNIT (AFBC/CTIU)**

**MORGANTOWN ENERGY RESEARCH CENTER**  
DOE - \$85,000  
3/76 - Continuing

**OBJECTIVES** – The immediate tasks for the stacked-bed AFBC/CTIU are to complete final design, obtain bids, and let contracts for long-lead equipment for the facility. The long-range objectives are to use the CTIU to test and evaluate improved solids-handling and fuel subsystems, develop the technology for vertical stacking of multiple beds, and serve as a boiler development laboratory to test tube bundle geometrics, component materials, instruments, and control concepts in operating fluidized-bed combustors. The CTIU will also provide design data for integration with commercial industry and utility AFBC applications.

**RECENT WORK AND ACCOMPLISHMENTS** – Preliminary design of the CTIU project has been completed and project approval obtained. Initial funding has been received and detail design begun. The design packages for the steam generator, building, and material handling systems have been completed and procurement packages issued. Site selection was finalized with West Virginia

University and a lease for the land use approved. A revised project scope limits the processing of one coal at a time.

**PLANS FOR THE COMING YEAR** – The procurement phase of the project will continue along with the start of actual construction at the site. The remainder of the major equipment procurement packages, including the control system, will be issued early in 1978.

### **ATMOSPHERIC FLUIDIZED-BED BENCH-SCALE STUDIES**

**MORGANTOWN ENERGY RESEARCH CENTER**

**DOE - \$697,000**

**6/75 - Continuing**

**OBJECTIVES** – This project is providing research support for the development of atmospheric fluidized-bed combustion (AFBC) technology. Specific studies are aimed at defining combustion, operating, and emissions characteristics of a wide variety of fuels; evaluating components and ancillary equipment in a hot-combustion environment; cold modeling studies of flow dynamics and mixing patterns as affected by feed-point location, bundle configuration, and other hardware variables; and small-scale studies supporting these aims.

**RECENT WORK AND ACCOMPLISHMENTS** – One of two 18-inch-diameter MERC combustors is dedicated to testing low-quality fuels including anthracite refuse, Montana subbituminous coal, Texas lignite, and lignite refuse. Defining operating and emissions characteristics of these fuels is helping to identify additional fuel reserves suitable for AFBC applications. The anthracite refuse studies, for example, have shown sufficient potential to develop a request for AFBC test-site proposals. Lignite and washed lignite refuse have shown promise of AFBC applications with reduced sorbent needs to meet existing SO<sub>2</sub> emissions controls. Limited tests have been carried out to define particulate emissions characteristics while burning subbituminous coal. Exploratory tests on a small 6-inch combustor have revealed promising novel ways to disengage carbon particles from exhaust gases and to increase carbon burnup efficiency.

**PLANS FOR THE COMING YEAR** – Investigation of combustion characteristics of low-quality fuels will continue with tests of oil shales, washed and unwashed high-sulfur western Kentucky coals, pyrolysis char, and high-sulfur heavy oil. A second 18-inch combustor will be commissioned and component equipment testing initiated. A 6-ft by 6-ft cold model will be installed and testing initiated to define effects of feed point locations, tube bundle configuration, and other hardware variables on solids flow patterns and mixing rates. Supporting small-scale studies in the 6-inch combustor will be tailored to speed testing in the larger facilities by screening potential test configurations.

### **AFBC DEMONSTRATION PLANT PROJECT**

**OAK RIDGE NATIONAL LABORATORY**

**DOE - \$300,000**

**7/15/77 - Continuing**

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technical feasibility, environmental acceptability, and overall economics while providing design data and operating experience for utility-size boilers using the AFBC process.

**RECENT WORK AND ACCOMPLISHMENTS** – A Program Opportunity Notice (PON) is being prepared by the Division of Power Systems at DOE to solicit proposals from prospective industrial partners for this cost-sharing project. The PON includes technical requirements, legal and procurement action guidance, proposal preparation instructions, and proposal evaluation criteria. The intent of the partnership arrangement as proposed in the PON is to provide a mutually beneficial cooperative agreement and working relationship between the DOE and the industrial partner to successfully demonstrate the commercialization potential of this new technology.

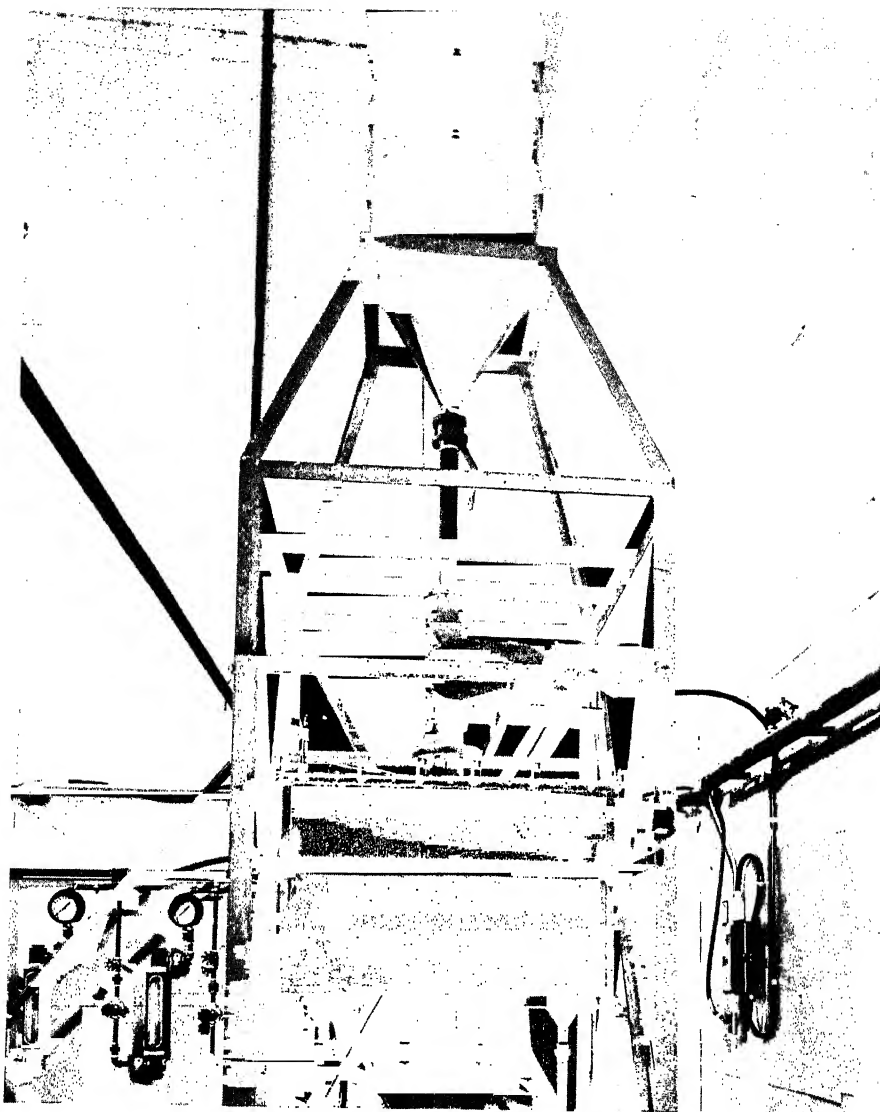
**PLANS FOR THE COMING YEAR** – ORNL will assist in the reevaluation of the solicitation document (PON).

### ATMOSPHERIC FLUIDIZED-BED COAL COMBUSTOR FOR COGENERATION

OAK RIDGE NATIONAL LABORATORY  
DOE - \$1,500,000  
1974 - Continuing

**OBJECTIVES** – This work involves developing the technology for a fluidized-bed coal combustion system as a source of high-temperature air for gas-turbine cogeneration plants to provide electric power and process heat for industrial applications. The program is directed toward the ultimate development of systems in the size range of 5 to 50 Mw<sub>e</sub>.

**RECENT WORK AND ACCOMPLISHMENTS** – The supplemental studies begun under the coal-fueled Modular Integrated Utility Systems (MIUS) Program were continued, the first tasks of the Coal Combustor for Cogeneration Program were initiated. The supplemental study tasks included tests on the coal-feed system, fluidized-bed heat transfer, and fireside corrosion of fluidized-bed cooling tubes. The coal feed system was operated continuously for 1000 hr without incident. A heat transfer test was run on an air-cooled tube in the Fluidyne Engineering Corporation fluidized-bed test combustor at a bed temperature of 900°C (1650°F) over a fluidizing velocity range of 0.25 to 0.65 m/sec (0.8-2.2 ft/sec) with a mean particle size of 460  $\mu$ m. The heat transfer coefficient was found to vary from 227 W/m<sup>2</sup>·°C (40 Btu/hr-ft<sup>2</sup>·°F) to 483 W/m<sup>2</sup>·°C (85 Btu/hr-ft<sup>2</sup>·°F). A corrosion test on air-cooled tubes made up of sections composed of 304, 310, 316 stainless steel, Inconel 600, and Incoloy 800 was run in the Fluidyne fluidized-bed combustor for a period of 500 hr. The tubes were maintained at a constant maximum wall temperature of about 870°C (1600°F) and then subjected to metallographic examination. The results were encouraging. No evidence of erosion was found, and a thin, hard scale composed of about 60 percent CaSO<sub>4</sub> was present on the tubes. No measurable loss of wall thickness was observed. A metal oxide layer of about 0.025 to 0.038 mm (0.001 to 0.0015 in.) was seen on each of the materials. The Incoloy 800 specimen showed intergranular oxidation attack for a depth of about 0.03 mm (0.0012 in.). No intergranular corrosion was observed in 304 and 310 stainless steel and Inconel 600. A second corrosion test of 1000-hr exposure was run. In two instances early in the test, Inconel 600 tubes failed and they were dropped from the test program. The 1000-hr test was completed without further incident.



*Coal Feed System Test Facility*

The Coal Combustor for Cogeneration Program was initiated in August 1977, and work was begun on two major tasks: preparation of performance specifications for use in obtaining conceptual design proposals from furnace manufacturing firms for both a 90-MW(t) combustor and a 1.5 MW(t) combustor test unit and performance of an assessment of the potential applications of gas turbine cogeneration systems for industrial use. The first draft of specifications was completed for review. A literature survey was done, and four sources that present a comprehensive survey of industrial energy use were found. A number of industrial processes that are well suited to the gas turbine cycle have been identified.

**PLANS FOR THE COMING YEAR** – The application assessment study will be completed. Detailed designs will be obtained for the test combustor, and a final design will be selected. Design work will proceed on the test system layout and the combustor support systems. Tests will be run with the coal-feed system feeding coal to the fluidized-bed cold flow model. A 3000-hr corrosion test will be run on the candidate tube materials.

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## PFBC COMPONENT TEST AND INTEGRATION UNIT (CTIU)

ARGONNE NATIONAL LABORATORY  
DOE - \$600,000  
7/1/75 - Continuing

**OBJECTIVES** – The CTIU is being used to: test and evaluate components, sampling, and instrumentation developments, control concepts and techniques, and materials of construction proposed for PFBC systems; have the flexibility to investigate alternative PFBC concepts; and have the capability for investigation of more advanced concepts of PFBC and limestone regeneration. The CTIU is a flexible test facility capable of investigating various pressurized fluid-bed concepts. Although the facility will not be used to produce electricity, the hot off-gas can be expanded through a turbine test cascade or, if necessary, a gas turbine to provide test data. The subsystems are also designed to allow for addition or modification of components to evaluate the performance of this equipment.

**RECENT WORK AND ACCOMPLISHMENTS** – A Comprehensive Analysis report was prepared, submitted to FE, and approved. All major design decisions were made and preliminary engineering completed. QA levels were defined. An exchange of information occurred between the CTIU and the IEA technical direction groups. A procurement for body flanges was let. An Industrial Review Board was organized and met. The Title I report is now being written and will be presented to DOE early in FY 1978.

**PLANS FOR THE COMING YEAR** – The Title I report will be submitted and the detailed design effort concluded. The bid packages for required procurements are to be completed. Construction is scheduled to begin at the end of FY 1978, with completion to be accomplished early in 1980.

## PFBC COMPONENT TEST AND INTEGRATION UNIT

STEARNS-ROGER ENGINEERING CO.  
DOE - \$20,000,000  
8/76 - 3/80

**OBJECTIVES** – This work is in support of the overall DOE program to develop pressurized fluidized-bed combustion technology for the environmentally clean utilization of the nation's coal reserves in the industrial and electric utility sectors of our economy. The CTIU will provide an experimental facility for integrated process and component development and testing of all aspects of PFBC technology.

**RECENT WORK AND ACCOMPLISHMENTS** – This CTIU work is technically directed by Argonne National Laboratory, where the facility will be built and whose personnel will operate it. Using the Argonne Conceptual Design which was published in May 1975, Stearns-Roger performed tradeoff studies and made an independent factored estimate of the facility. This information was integrated with the conceptual design and presented to ERDA-FE by Argonne in February 1977, in the form of a Comprehensive Analysis. At this time, approval was received to proceed with preliminary engineering leading to a Title I Report for presentation to DOE in early 1978.

The key component of the PFBC/CTIU is the pressurized combustor. To assure flexibility for testing various PFBC concepts, the combustor pressure containment vessel shell contains a set of

body flanges near its top, permitting the disassembly of the shell so that various internal components may be placed inside, in a modular fashion, for testing. A design philosophy based on replaceability and expansion has been applied throughout the CTIU plant to facilitate the testing of various equipment components associated with PFBC.

**PLANS FOR THE COMING YEAR** – Preliminary engineering will be completed and a Title I report presented to DOE. Packages for required procurements will be completed and contractor(s) selected.

## **PFBC COMBINED-CYCLE POWER PLANT CONCEPTUAL DESIGN**

**BURNS AND ROE INDUSTRIAL SERVICES CORPORATION**

**DOE - \$1,635,052**

**6/76 - 6/78**

**OBJECTIVES** – This study is designed to prepare a conceptual design for an electric power generating plant that will directly combust high-sulfur coal in a pressurized, fluidized-bed combustor in conjunction with a combined gas/steam-turbine cycle to generate electric power in an environmentally acceptable manner.

**RECENT WORK AND ACCOMPLISHMENTS** – A project team was assembled to conduct the engineering evaluation of the PFBC Combined-Cycle Power Plant Study. It is composed of Burns and Roe as prime contractor with Babcock & Wilcox and United Technologies as subcontractors. The size and operating mode were established: a power rating of 500 to 600 Mw<sub>e</sub> and base loaded to optimize performance; however, modularity and turndown capability are primary considerations in the study activities. Performance calculations were made for various cycle configurations such as PFB fired gas turbine with: (a) a waste heat boiler, (b) exhaust firing in an AFB boiler, (c) reheat at power turbine inlet and a waste heat boiler, and (d) reheat at low turbine inlet in conjunction with exhaust firing in an AFB boiler. For each of these cycles, a preliminary screening capital cost and operating cost estimate was made. As a result of the performance data and the limited economic studies, Cycle (b) was selected for investigation. This plant is based upon a combined gas-turbine (UTC's FT-50 Engine) and steam-turbine cycle. The gas-turbine combustor is an air-cooled pressurized-fluidized bed designed for coal firing. Exhaust from the gas turbine is fired in an atmospheric fluidized-bed boiler, which generates superheated steam from the "bottom" portion of the cycle. A preliminary estimate of the capital and operating costs, expressed in mid-1977 dollars, for the 600-Mw<sub>e</sub> commercial plant has been developed. A capital cost of \$334,000,000 or \$580/kw of net capacity and a cost of electricity of 28.9 mils/kwh were obtained. While the numbers presented will probably be refined somewhat as the project continues, no major changes are anticipated.

**PLANS FOR THE COMING YEAR** – The current contract is scheduled for completion in June 1978. Work will continue in the following areas: refinement of cost data, identification of developmental areas, determination of environmental impact, investigation of alternative cycles, and reliability and maintainability analysis.

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## PFB PILOT PLANT PROGRAM

CURTISS-WRIGHT CORPORATION  
DOE - \$30,300,000; CW - \$4,400,000  
3/76 - 5/82

**OBJECTIVES** – This PFB program involves the design, construction, and operation of a coal-fired gas turbine pilot electric plant to evaluate the PFBC process as a means for production of electric power from combustion of high-sulfur coal. Additional objectives include the conceptual design of a PFB commercial coal-fired 300-to-500-Mw combined-cycle generating station to provide a recommended pilot plant concept; pilot plant site evaluation and environmental assessment; technology support development programs, which provide data on the performance of the selected PFB design; and reassessment of the commercial plant conceptual design after the PFB pilot plant operating period. The PFB combustion process applied here involves the combustion of coal in the presence of a sulfur sorbent material at a temperature below 1750°F. Compressed air from a gas turbine is used in part for combustion and the remainder is indirectly heated in a tubular heat exchanger within the fluidized bed. The pilot plant, which will produce an equivalent of 13 Mw<sub>e</sub>, will address a number of key technical issues. Durability, maintenance and reliability of the PFB and turbine as well as environmental performance will be evaluated. The program will provide design data to verify scaleup of the PFB units from about 42 percent of commercial plant size for the electric utility industry.

**RECENT WORK AND ACCOMPLISHMENTS** – The conceptual design and cost analysis of the PFB 500-Mw commercial electric power generation plant was completed. The entire station includes three 100-Mw gas turbine double-ender modules and one 180-Mw steam turbine. Performance analyses indicate a wide range of power-generation turndown flexibility while maintaining low plant heat rate. The PFB pilot plant preliminary design has been completed and is representative of the commercial design concept. The pilot plant will be located at the Curtiss-Wright Wood-Ridge, New Jersey facility where an existing 7-Mw Gas Turbine Total Energy Power Plant, using distillate fuel or natural gas, has been in operation since 1970. The PFB pilot plant design makes use of this existing power plant with considerable incorporation of currently operating equipment. The pilot plant will operate with a coal flow rate of about 5 t/hr. The gas turbine will produce over 7 Mw of electric power and the waste heat recovery boiler will produce 58,000 pph of steam providing the equivalent power for the balance of the 13-Mw plant. The pilot plant is designed for ease of maintenance and component servicing and replacement that would be expected in a pilot test program. Space is provided for alternate types of equipment for future test programs. The final design of the PFB pilot plant was initiated in September 1977 and will be completed in the second quarter of 1978.

The site evaluation and environmental assessment for the pilot plant have been completed including preparation and compilation of the following: real estate report, site master plan, climatological and meteorological data, foundation investigation and soil analysis, site and local resources survey, site transportation study, and environmental impacts discussion. Technology support tests have been conducted to evaluate PFB air-cooled heat-exchanger performance, and heat-exchanger and turbine materials corrosion and erosion resistance in simulated PFB systems environments. Testing for heat-transfer characteristics of vertical finned tubes in the fluidized bed was conducted initially in a coal-fired 12-in-diameter bed to determine the effect of number of fins, fin height, and bed particle size on heat transfer coefficient and combustion efficiency. Additional



testing for heat-exchanger performance was conducted in a 2-by-3-ft pressurized fluid bed 8 ft in height. Heat-exchanger finned tubes using candidate alloys were tested for over 4300 hr in a commercial fluid bed used for limestone calcination and about 670 hr in a commercial fluid bed for sewage sludge incineration. These tests were directed toward obtaining comparisons of the resistance to corrosion-erosion attack of candidate alloys under long-term exposure in an aggressive environment. Further testing on both heat-exchanger tube and turbine blade materials was conducted in laboratory rigs under accelerated exposure conditions. A small gas turbine/pressurized fluidized bed (SGT/PFB) technology rig has been designed, constructed, and erected. Development testing on coal was initiated in midyear to define operating parameters and long-term materials durability in a PFB environment.

**PLANS FOR THE COMING YEAR** – The PFB pilot plant final design will be completed, including detailed construction plans and cost estimates and plant construction bid package preparation, bid evaluation, and recommendations for award. Technology support testing and long-term materials evaluation using the SGT/PFB technology unit will be completed. Pilot plant long-term procurement and pilot plant construction will be initiated.

#### GRIMETHORPE EXPERIMENTAL FACILITY

NATIONAL COAL BOARD (IEA SERVICES) LTD.  
DOE - \$8,123,000; National Coal Board - \$8,123,000  
7/28/75 - 2/23/79

**OBJECTIVES** – This project's goals are broadly threefold: first, to build an experimental facility to study combustion, heat transfer, gas cleanup, corrosion and energy recovery in a pressurized fluidized-bed combustion system; second, to carry out tests over a wide range of operating conditions and to make measurements in greater detail than would be either economical or possible in an integrated unit; and third, to provide data for the analytical modeling of design data for a larger commercial plant by empirical data extrapolation. The experimental facility provides a means to conduct research into a number of aspects of large commercial plants, including the effects of high ash coal on combustor performance, the effect of dolomite additives on sulphur retention in addition to a means of studying heat transfer coefficients, heat transfer element materials, and geometry.

**RECENT WORK AND ACCOMPLISHMENTS** – All of the contracts for the design and fabrication of the principal plant items have been let, and much of the detail design work on this plant is now complete. Fabrication of the turbocompressor and combustor has begun. A number of auxiliary items including the pipework, control system, and chimney contracts have recently been let and design work has started. At the Grimethorpe site, civil work is underway and on schedule for the main and control building foundations with the piling well advanced.

**PLANS FOR THE COMING YEAR** – Completion of the main building, delivery, erection, installation, and interconnection of the principal plant items is planned in preparation for commission and test in early 1979.

# COAL-FIRED COMBINED-CYCLE DEVELOPMENT PROGRAM

GENERAL ELECTRIC COMPANY

DOE -\$4,773,396

6/76 - 6/78

**OBJECTIVES** – The coal-fired combined-cycle (CFCC) power plant conceptual design is being evaluated, utilizing pressurized fluidized-bed combustion (PFBC), and a supporting technology development program is being conducted. The CFCC approach provides cooling of the fluidized bed by steam tubes that supply steam to a conventional steam-turbine generator. The pressurized combustion gases drive a gas turbine generator. The low combustion temperature (1750°F or below) reduces the  $\text{NO}_x$  concentration and the dolomite bed material absorbs sulfur to limit  $\text{SO}_2$  emission. These features allow a wide range of low-quality coal to be burned economically and efficiently.

**RECENT WORK AND ACCOMPLISHMENTS** – The commercial PFB power plant conceptual design study has been largely completed. The design features a single steam turbine-generator producing 493  $\text{Mw}_e$  and three 153  $\text{Mw}_e$  gas turbine-generators for a net plant output of 925  $\text{Mw}_e$ . The three combustor steam-generators operate at 10 atm, 1750°F with 4.5 ft/sec fluidizing velocity and 20 percent excess air, and provide better than 99 percent combustion efficiency. Supercritical steam generation is at 3500 psi, 1000°F with 1000°F reheat. The projected coal-pile to bus-bar plant efficiency is 40.3 percent. The selection of operating conditions has been based on cycle optimization studies and on the results of the technology development program. The combustor-steam generator design has been selected based on test results from the 2-ft-by-3-ft PFBC at NCB/CURL, Leatherhead, England. The waterwall combustor has been designed with four separate beds. The first three, operated in series, provide superheated steam, while the fourth is for reheat. Combustion efficiency in excess of 99 percent is achieved without a separate carbon burnup cell. Alternate combustor configurations, including once-through and refractory-lined boilers, have been investigated.

A major technology issue in PFB combustion of coal is corrosion of gas-turbine hot parts by alkali metal sulfates in the combustion gases. Combustion chemistry and thermodynamic considerations have demonstrated that there are no bed additives that could suppress alkali metal vapors sufficiently to limit hot corrosion; therefore, to maintain combustion temperature (and cycle efficiency) at the highest practical level, new alloys are sought to serve as corrosion-resistant claddings for high-strength turbine blade materials. A 1500-hr bench-scale hot-corrosion screening test of fourteen potential alloys was performed in a small burner rig using fuel doped with sodium, potassium, and sulfur. Of these materials, 11 are being subjected to long-term test and have accumulated over 2000 hr at 1600°F. Both FeCrAlY and CoCrAlY alloys continue to look promising for the PFBC application. Development of the cladding process has continued. The properties of 11 potential cladding materials have been studied as a function of annealing temperature to maximize room temperature ductility and formability to 10-mil sheet. New alloys have been identified that show significant potential. Considerable attention has been devoted to design of the bucket airfoil template and fabrication of the cladding to avoid wrinkles and improve the fit over the substrate prior to bonding. Cold forming of the platform cladding and activated diffusion bonding of the tip caps have been demonstrated. The mechanical properties of the cladding/substrate combination have been measured, including tensile strength, creep rupture, and high- and low-cycle fatigue. Clad airfoil specimens were fabricated and supplied to Exxon Research and Engineering for test in a related DOE program.

A second major technology issue in the PFBC system is removal of ash and bed particles from the combustion gas to protect the gas turbine from erosion. Studies to characterize the PFB efflux and the tolerance of the turbine to erosion have been carried out. The results of these studies define the particulate removal efficiency required for the hot-gas cleanup system. The reference plant design includes a granular bed filter for positive particulate control; however, studies have shown that improved inertial separators might also provide adequate erosion protection at considerably lower cost. Experiments to evaluate and improve cyclone separators have demonstrated the importance of electrostatic effects, which if understood and optimized could lead to significant improvements in cyclone performance. Additional experiments have demonstrated that the performance of conventional cyclones is limited by short-circuiting of the dusty inlet flow into the outlet. Methods to control such short-circuiting are being investigated.

**PLANS FOR THE COMING YEAR** — A major design review of the reference commercial plant will be held, with DOE, electric utility, and industry participation. More definitive estimates for both installed capital cost and operating cost will be prepared based on existing detailed equipment lists. Combustor steam-generator performance and PFB efflux estimates will be confirmed and Aerodyne cyclone hot testing will be accomplished during testing at NCB/CURL. These tests, as well as the Fireside II program at the Exxon mini-plant, will include further exposure of candidate materials to PFB combustion gases. Results of these exposures will be evaluated and analyzed. A final report and other contractual documentation will be completed.

## **MODELING OF FLUIDIZED-BED COMBUSTION OF COAL**

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

DOE - \$955,247

5/1/76 - 1/31/78

**OBJECTIVES** — MIT has organized a broad-based multidisciplinary group to develop a system model for using FBC technology commercially. The overall objectives of the program are to establish a comprehensive system model precise enough for process and engineering design optimization and a data base system that will reposit all relevant data on coal-based FBC in a single site that can be used to answer various queries from remote sites; and to document information gathered or generated in the foregoing tasks in a well-organized readily usable form. The 21-month initial phase is focused on developing a model system using state-of-the-art information and the establishing of a data base system accessible to remote site users. A planning document will also be developed identifying information gaps in either the modeling or data base task, and recommending ways in which this information may be obtained most effectively.

**RECENT WORK AND ACCOMPLISHMENTS** — Modeling studies have revealed that physical phenomena involved in an FBC system are unique, differing significantly from those prescribing a

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combustion, CO burnout, and NO<sub>x</sub> emission are being developed. The slow-bubble and char-combustion chemical kinetics models have been completed and incorporated into the system model resulting in an initial version of a refined first-generation model. Without these and projected improvements, the model would not be precise enough for engineering use. In data base system work, the nine most established systems were studied, and System 204 selected. A prototype data base system has been implemented using an MIT-developed RDMS system. A core selection of literature data was recorded in the prototype system and subsequently machine transferred to the 204 system. A text file for preserving the important attributes of the papers and six example applications have been developed. The data base will soon be operational, although it will have only a relatively limited amount of data repositied in it. The needed information for the planning document is being gathered.

**PLANS FOR THE COMING YEAR** – The tasks to be performed include development of: a second-generation system model that will have a number of key features beyond the state-of-the-art and specifically developed for this application; 20 ongoing key feature models, with another 20 to be added; the data base management system which will be rendered fully operational, including modifications of data structure design, improvements in file organization, query optimization, enhancement in user interface capabilities, and promotion of the concept of using data base as a monitoring tool for DOE contractors who are conducting experiments to gather better and more complete data; and a network of information to be used as a basis for a more refined future planning document.

## SUPPORTING STUDIES IN FLUIDIZED-BED COMBUSTION

ARGONNE NATIONAL LABORATORY  
DOE - \$1,150,000; EPA - \$325,000  
1968 - Continuing

**OBJECTIVES** – Because fluidized-bed combustion appears to offer cost and efficiency advantages over other methods for burning coal in an environmentally acceptable manner, DOE is developing and demonstrating the feasibility of a fluidized-bed combustion process for power and industrial heat applications. The aim of the Argonne National Laboratory program is to conduct supporting R&D on problems related to pressurized combustion processes and sorbent regeneration in order to: decrease the quantity of solid waste discarded from the combustion system by (a) developing an economical process for regenerating the sulfated limestone, (b) developing methods for identifying the more reactive limestone and dolomites, (c) modifying the structure of limestones physically or chemically to obtain a more reactive stone; determine the extent that the alkali metal compounds are transported in the flue gas and evaluate methods for reducing this; evaluate methods for reducing the concentration in hot flue gas of particles of a size that erodes turbine blade metal; and develop and test prototype on-line analytical instruments.

**RECENT WORK AND ACCOMPLISHMENTS** – The behavior of Greer Limestone has been extensively studied because the stone will probably be used in the 30-Mw Rivesville, West Virginia combustor. Sulfated Greer limestone can be effectively regenerated to CaO by reductively decomposing it at 1100°C and ~1.5 atm. Partial combustion of coal supplies the heat and the required reducing gases. Extent of regeneration improved with higher temperature and longer particle residence time. The reaction rate at 1100°C is high and only about 7 minutes residence time is required for effective regeneration; SO<sub>2</sub> concentrations in the off-gas are ~10 percent, sufficiently

high for recovering sulfur values using commercially available processes. Processing Greer limestone through ten combustion-regeneration cycles demonstrated that the quantity of new stone required in the process can be reduced significantly if the sulfated stone is regenerated. The reactivity of the stone decreased with increased cyclic use probably because of changes in particle porosity, and the extent of regeneration remained acceptable in all cycles. Mass and energy balance flow-sheets have been prepared for combustion-regeneration systems up to 800-Mw<sub>e</sub> size.

Since the cost of operation and the environmental impact of the FBC plant depends on the quantity of limestone that must be used, a study is underway on methods of predicting the performance of different limestones in large fluidized-bed combustors and methods of increasing the SO<sub>2</sub> capacity of limestones with poor performance. It has been found that the performance of limestones in fluidized-bed combustors can be predicted from kinetic data on the reactivity with SO<sub>2</sub> measured using a thermogravimetric analyzer. The optimum SO<sub>2</sub> reactivity of calcitic limestone is obtained when the average diameter of the pores in the calcined limestone particles lies in the range 0.3 to 0.4  $\mu$ m. The average pore diameter of limestones can be increased by several physical and chemical treatments.

A potential problem when using pressurized fluidized-bed combustors to generate hot high-pressure gas for gas turbines is the possibility that the alkali metals present in the coal and limestone might be transferred to the turbine and form alkali sulfate films that would lead to hot corrosion. Studies are underway to determine the extent that the alkali metals are transported from the reactor to the turbine and to investigate methods for the reduction of this transport. The emission of alkali metal compounds from coals with high ash contents is not as large as from low ash coals. It was found that if hot flue gas containing gaseous alkali metal compounds (viz. gaseous NaCl) is passed through hot filters composed of various clay materials, significant removal of the alkali metal compounds is achieved. Filtration studies, using a fixed bed of granular limestone, have demonstrated that relatively good filtration efficiency is obtained for particles of a size that can damage turbine blade metal. This suggests that by the proper choice of materials it may be possible to combine the removal of particulates and gaseous alkali metal corrodents in one device. Two prototypes of on-line, light-scattering particle-size analyzers were tested and evaluated.

***PLANS FOR THE COMING YEAR*** — Regeneration studies will be continued to investigate processes other than reductive decomposition and equipment other than a fluidized-bed unit to obtain higher SO<sub>2</sub> concentrations in the off-gas, higher particle reactivity after regeneration, or simpler operation; provide flowsheet and cost information for regeneration units attached to 800-Mw combustors; determine if regeneration is economically effective when compared with once-through use of limestone; improve the reductive-decomposition regeneration process; determine the behavior of trace elements; evaluate the cyclic behavior of other limestones; and evaluate sulfur recovery processes for removing SO<sub>2</sub> from the regenerator off-gas and select one for further study.

potentially corrosive alkali metals in pressurized combustion will be examined further, and instrumentation for on-line detection and analysis of alkalis in the flue gas stream will be evaluated.

## **GRANULAR-BED FILTER DEVELOPMENT PROGRAM**

**COMBUSTION POWER COMPANY, INC.**  
**DOE - \$928,306**  
**1/24/77 - 1/23/78**

**OBJECTIVES** — The effectiveness of a moving bed filter in removing particulates from the hot gases produced by a pressurized fluid-bed combustor is being investigated. The development of efficient high-temperature gas cleanup devices is required for energy conversion cycles in which the gases are expanded through gas turbines and in other cycles where corrosion, erosion, and deposition caused by particulates cannot be tolerated. This contract covers Phase I of the program and includes parametric cold-flow testing of an annular 1500-acf/m in (100-ft/m in approach velocity) filter at atmospheric pressure, the development of a companion mathematical model, and the correlation analysis of the model predictions with the experimental results. The objective of Phase I is to acquire a better understanding of the filtration mechanisms and through the development and correlation of a mathematical model to prepare for subsequent hot-flow experiments.

**RECENT WORK AND ACCOMPLISHMENTS** — The parametric test program and the development of a mathematical model were completed. A parametric test matrix was developed from latin squares, and 121 tests were conducted in which the independent variables were gas flowrate, particulate concentration and size distribution, media size and flowrate, and active filter thickness and height. The measured dependent variables were filter-pressure drop and outlet particulate concentration and size distribution. The mathematical model was defined for total and fractional efficiencies for the filtration mechanisms of impaction, interception, diffusion, and sedimentation using the theoretical and experimental work from the literature as the basis for the efficiency equations. The equation coefficients were adjusted by the results of experimental data obtained in the parametric tests. Correlation of the mathematical model predictions with the experimental results is in process.

**PLANS FOR THE COMING YEAR** — The correlation of the model with the test data and the final report for Phase I will be completed. Phase II will be conducted, which includes hot-flow testing in a 1500-acf/m filter of the same geometry as the cold-flow filter. Coal, sorbent, and corrosion inhibitors will be used with fluid-bed combustion to produce the particulate-laden hot gases at 1600°F, 1.5 atmospheres. Effects of filtration adequacy will be determined from a cascade of turbine blades downstream of the filter. The mathematical model will be updated to reflect the influence of temperature and the properties of real ash.

## **AGRICULTURAL UTILIZATION STUDIES: FBC BYPRODUCT MATERIAL**

**DEPARTMENT OF AGRICULTURE**  
**DOE - \$1,750,000**  
**7/1/76 - 6/30/81**

**OBJECTIVES** — Evaluation is being made of the solid wastes from fluidized-bed combustion for potential use in agriculture. The work involves detailed chemical and physical analysis of the material followed by greenhouse, growth chamber, and field studies.

**RECENT WORK AND ACCOMPLISHMENTS** – Chemical characterization of various batches of FBM byproduct material indicate tremendous variation in the concentration of most elements. Elemental composition of fluidized-bed material will vary considerably between different size fractions. At Morgantown, West Virginia, studies were conducted comparing FBM with agricultural lime plus equivalent sulfur and with fly ash. Buckwheat and sweet clover were used in the test crops on a low pH strip mine spoil. These studies have been harvested. Growth and nutrient uptake of soybeans, oats, tall fescue, swiss chard, red clover, and buckwheat are being compared on an acid soil after treatment with FBM, and lime and sulfur sources. At Beltsville, Maryland, studies were initiated to determine effects of different amounts of FBM on root-shoot growth of seedling apples and peach trees on widely different soils. Rates of FBM and rock phosphate washer rejects were compared alone and in combination for their effect on yield of weeping lovegrass and birdsfoot trefoil grown on an acid stripmine spoil. A combination of FBM and phosphate washer rejects produced highest yields with both weeping lovegrass and birdsfoot trefoil. The spoil pH was raised from 4.1 to 4.6 by 16 tons of FBM. Greenhouse studies on sand and gravel spoil were conducted to compare growth of tall fescue after treatment with several rates of FBM and rates of dolomite. Fescue yields were the same at rates below 10 t/acre, but yields from dolomite were higher at 15 to 20 t/acre of each. Greenhouse plot studies were initiated at Byron, Georgia, to determine effect of FBM as compared to calcium sulfate and calcitic lime on seedling growth of peaches and pecans.

Studies with alfalfa and corn were started in 1976 in Pennsylvania, at four locations with widely different climate and soil types. In West Virginia, field studies using tomatoes and corn as test crops and comparing FBM with lime, and lime plus sulfur at equivalent rates of FBM are in their second year. First-year results indicate no significant effect on sweet corn yields but a slight reduction in tomato yields when using FBM. Chemical analysis of tomato fruits indicate that FBM produced a slight increase in the zinc, boron, chromium, and nickel levels; however, all these levels were well within acceptable range for human consumption. Field studies were also initiated during 1976 and 1977 on low pH strip-mine spoils in West Virginia and Maryland, to determine the potential of using FBM for production of forage species. Preliminary results indicate that FBM had greater liming potential when used in combination with an organic waste material such as composted garbage or sewage sludge. Field studies were initiated during the spring of 1977 at Georgetown, Delaware, using corn as a test crop and comparing rates of FBM with dolomitic limestone.

**PLANS FOR THE COMING YEAR** – Forage samples will be analyzed for nutritive value, and plots will be harvested for yield determination during 1977. Grain and forage will be collected from the Georgetown corn study and analyzed for quality components. Grain will be used in feeding trials with baby chicks at the U.S. Poultry Lab in Georgetown. Preliminary feeding trials will be initiated using FBM as a part of the feed mixture.

## EVALUATION OF SULFATE-BEARING BYPRODUCTS FROM FBC

MIDWEST RESEARCH INSTITUTE

DOE - \$42,750\*

12/20/76 - 11/30/77

**OBJECTIVES** – This study is evaluating the effectiveness of sulfate byproducts from fluidized-bed combustion of coal for improving the engineering properties of fine-grained soil. The effects will be

\*This study was conducted under a modification-extension to Federal Highway Administration Contract DOT-FH-11-8515, "Use of Waste Sulfate for Remedial Treatment of Soils." Funds were provided by DOE under Interagency Agreement No. E(49-18)2491 with the U.S. Department of Transportation.



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determined of sulfate byproducts on the unconfined compressive strength, wet-dry or freeze-thaw durability and volumetric stability of 15 fine-grained soils indigenous to proposed sites of fluidized-bed generating plants. It will also include evaluation of the effects of additives (such as hydrated lime, fly ash, and cement kiln dust) to enhance the beneficiation of soil engineering properties of the soil-sulfate byproduct system.

**RECENT WORK AND ACCOMPLISHMENTS** – A laboratory study has been completed in which spent-bed material (SBM) from a fluidized-bed coal combustion pilot plant in Alexandria, Virginia, was mixed with 15 fine-grained soils. The soils used in the study were indigenous or similar to those indigenous to proposed fluidized-bed facilities in Illinois, Indiana, Missouri, Michigan, Ohio, Pennsylvania, West Virginia, and Kentucky. Results indicate that SBM is an excellent soil stabilizer equivalent in effectiveness with hydrated lime.

**PLANS FOR THE COMING YEAR** – The final report for the study will be reproduced and distributed by the Federal Highway Administration.

### POTENTIAL USES FOR RESIDUE FROM FBC PROCESSES

L. JOHN MINNICK CONSULTANT  
DOE - \$259,530  
11/76 - 11/78

**OBJECTIVES** – This study is evaluating the solid byproducts from various fluidized-bed combustion (FBC) boilers in terms of potential commercial applications for the byproduct materials, or certain components of the materials. In addition, the investigation will explore methods by which they can be safely disposed of should commercial applications be slow in developing or of insufficient magnitude to account for their entire use.

**RECENT WORK AND ACCOMPLISHMENTS** – Representative samples of spent-bed material have been obtained from atmospheric FBC boiler units in Alexandria, Virginia; Rivesville, West Virginia; and Minneapolis, Minnesota. Samples of FBC fly ash have also been obtained from the Alexandria plant. These materials have been thoroughly analyzed in the laboratory to determine their physical and engineering properties, as well as their chemical compositions. Other characteristics of these byproducts, including their lime content, reactivity, and cementitious properties, have been evaluated. Several techniques, such as magnetic separation, wet tabling, dry tabling, air classification, milling, and hydration, have been used to beneficiate, concentrate, or in some way separate various components from the FBC residue and fly ash materials. The relative success has varied; so far, it appears that a combination of milling or particle-size reduction in wet slurry form, followed by wet tabling, produces the most promising separation products. Many commercial applications have been investigated in the laboratory, and several of these have potential including the use of the spent bed material, or portions thereof, as a component of stabilized road base mixtures; structural fill material; a stabilizing agent for plastic soils, coal mine refuse, and domestic or industrial sludge byproducts; possible raw material for the manufacture of concrete masonry units, sintered aggregate, and other structural products; an aggregate for use in asphalt paving mixtures; a neutralizing agent in water and waste water treatment; and a sorbent material for use in lime scrubber applications. A small-scale field demonstration project has been completed to evaluate the use of FBC residue in a pilot gas scrubbing system.



Disposal studies have focused on the application of proposed ASTM laboratory testing procedures to evaluate the runoff characteristics and leachate quality associated with placement of FBC residue in a landfill type of environment. This evaluation is considering the FBC residue in its "as received" condition, as well as beneficiated and stabilized FBC materials. Reef blocks have also been fabricated of FBC residue and are being studied in a salt water environment off the coast of Long Island to evaluate the possibility of ocean disposal of these materials.

**PLANS FOR THE COMING YEAR** – Work will continue on laboratory evaluation and characterization of FBC byproducts from a number of other FBC boiler units representing different operating conditions and diverse sources of bed material and coal. More commercially applicable beneficiation or separation processes will be investigated and the products analyzed. Commercial application studies in the laboratory will continue using all available FBC byproducts, as well as the potentially useful components separated from the various beneficiation processes. Small-scale field demonstrations are being arranged to evaluate FBC materials in stabilized road base and acid mine drainage treatment applications. The production of concrete masonry units using FBC residue in an actual plant operation will be investigated. Further extension of effort will be applied in the study of disposal effects of FBC byproducts, both by landfilling and ocean disposal. A control stockpile and simulated landfill model of FBC residue are also planned to more closely monitor weathering and field leaching of these materials.

## FLUIDIZED-BED BYPRODUCTS AS A SOURCE OF NUTRIENTS AND LIME

TENNESSEE VALLEY AUTHORITY  
DOE - \$29,000; TVA - \$14,500  
4/1/76 - 1/31/78

**OBJECTIVES** – This research seeks to determine the effectiveness of fluidized-bed materials (FBM) from FBC boilers as a source of nutrients for crops and as a liming material for acid soils and acid coal-mine spoils.

**RECENT WORK AND ACCOMPLISHMENTS** – Results obtained in greenhouse pot and laboratory experiments at Muscle Shoals, Alabama, show that FBM is a satisfactory source of sulfur for corn and peanuts, and as a soil liming material. High disposal rates of FBM initially were toxic, probably because of its high alkalinity (high-oxide content). Granular ( $< 3$  mm) and fine FBM were 8 and 47 percent as effective as fine  $\text{CaCO}_3$  for neutralizing acidity in an acid silt loam soil over a 5-week period. On pyritic acid coal mine spoil, lower liming rates did not affect spoil pH, but higher rates of granular FBM were 44 percent as effective as fine  $\text{CaCO}_3$  for increasing spoil pH over a 10-month period. A field experiment on alfalfa is in progress at Muscle Shoals, Alabama, to compare FBM, limestone scrubber slurry and ground limestone. This evaluation will require 3 or more years. Three field experiments on peanuts in South Alabama initiated in 1976 are in progress in cooperation with Auburn University. The final crop year for these experiments is 1978.

**PLANS FOR THE COMING YEAR** – A few additional pot and laboratory experiments will be conducted but field experiments to study problems associated with large disposal rates of application are the primary need. The field experiments at Muscle Shoals and in South Alabama will be continued in 1978. Disposal of FBM on agricultural soils, coal mine spoils, or in dumping areas may give rise to pollution of surface water and groundwater. Some leaching studies in soil leaching

columns or lysimeters are planned. Large-scale field studies of FBM problems have been awaiting an adequate supply of FBM. Timing and costs of the suggested experiments, especially field experiments, will be substantially influenced by source and supply of FBM.

## UTILIZATION OF COAL CONVERSION PROCESS BYPRODUCTS

IIT RESEARCH INSTITUTE

DOE - \$264,062

6/28/74 - 9/27/77

**OBJECTIVES** – Economically viable uses of the byproducts of coal conversion processes (CCP) are being defined. Of the varying amounts of byproducts formed when coal is converted to clean fuels (solids, liquids, and gases), the chars and heavy tar residues have the greatest potential for utilization. Work undertaken at IITRI has been directed toward converting these residues to commercially useful materials and assessing the economic feasibility of CCP byproduct utilization. The purpose of the work is to convert these byproducts to benzene polycarboxylic acids with values greater than \$0.25 per pound. The utilization of CCP byproducts is expected to improve the overall economic feasibility of coal conversion and to reduce environmental problems associated with byproduct disposal.

**RECENT WORK AND ACCOMPLISHMENTS** – The work performed was aimed at determining the feasibility of converting the acid mixture obtained in the oxidation of coal conversion process byproducts to one or more commercially useful materials. One chemical process investigated was designed to convert the oxidation mixture to terephthalic acid by means of a modified Henkel reaction. A preliminary economic analysis showed that the process could be economically feasible if the yields were increased. The possibility of using a mixture of higher benzene carboxylic acids (CA's) as builders in detergent formulations was examined, but results were not entirely promising. During the final phase of the program, the feasibility of synthesizing low-cost thermosetting polymers from the carboxylic acid mixture obtained in the oxidation of the coal conversion process chars was investigated. The criteria utilized in the selection of polymerization approaches are: (1) the polymerization process should be commercially feasible; (2) the reactant(s) should be low-cost, readily available materials; (3) the polymerization should be relatively insensitive to the composition of the coal acid mixture, which may vary from batch to batch; and (4) coal acids should be an essential constituent of the final product. The synthesis of polyester resins was found to offer considerable promise. Among the various glycols investigated, ethylene glycol "bottoms," a byproduct of the synthesis of ethylene glycol, was found to be a useful low-cost co-reactant for polyesterification. Plastic sheets and glass reinforced composite structures were fabricated and tested. Very encouraging results have been obtained, which demonstrate the feasibility of manufacturing commercially useful polymers from CCP byproducts raw materials.

**PLANS FOR THE COMING YEAR** – Project work was completed in September 1977.

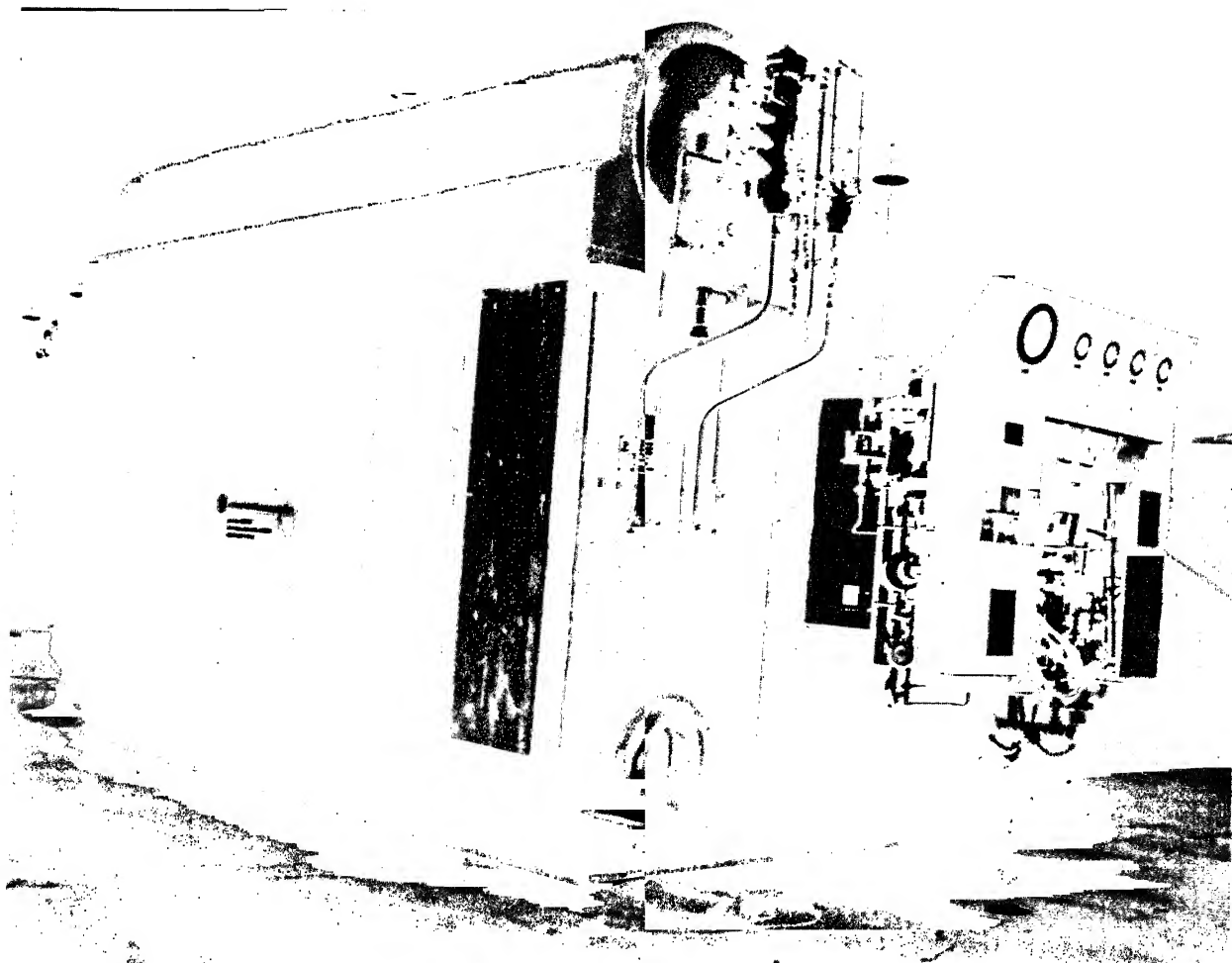
# COMBUSTION TEST FACILITIES FOR COAL-OIL SLURRIES

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$1,119,500

1966 - Continuing

**OBJECTIVES** – To demonstrate by long-term combustion tests the potential for using coal-oil slurries in industrial oil-fired boilers to extend U.S. oil supplies and reduce dependency on foreign oil, experimental work is underway in a 100-hp oil-fired firetube boiler. This work will be broadened after completion of a new facility centered around a 700-hp watertube boiler, which is representative of large industrial boilers. A secondary objective is the evaluation of coal-derived liquid fuels such as Synthoil, SRC-II, H-coal oil, and shale oil. Oil consumption could be reduced by 1 million bbl/d if all the nation's oil-fired boilers were fueled with a 40 percent coal-oil slurry. The objectives for the 500-lb/hr pulverized-coal-fired facility is to determine the handling, pulverizing, burning, and fouling characteristics of coal-derived fuels such as solid SRC and chars from the various gasification processes under development. Coal-derived fuels can be produced with low ash and sulfur contents, thus eliminating the most objectionable aspects of coal utilization in electric power plants and industrial boilers.



*A 700-HP Watertube Boiler, Representative Of Large Industrial Boilers, Being Installed at PERC for Coal-Oil Slurry Combustion Tests*

**RECENT WORK AND ACCOMPLISHMENTS** – The 100-hp firetube boiler gave good combustion performance with oil and 20 and 30 percent coal-oil slurries. The boiler was operated at full-rated capacity with both 20 and 30 percent coal-oil slurries without any decrease in boiler efficiency. A comprehensive experimental program oriented toward studying combustion characteristics of coal-oil slurries compared with No. 6 oil is underway. Combustion aerodynamics and radiative heat-transfer characteristics in the combustion chamber are being examined in detail. A limited number of combustion tests were also conducted with alternate fuels such as char-oil slurry and shale oil. The detailed design of a sophisticated slurry-combustion test facility, centered around a 700-hp (210 gal slurry/hr) watertube boiler, has been completed, and most of the construction contracts have been signed. The boiler will be completely instrumented and equipped with a computer-based data-acquisition system.

A Rheology Laboratory was established to determine density, viscosity, and settling characteristics for slurries made with different coals, coal concentrations and sizes, and stabilizing additives as a function of temperature and oil type. Data from this laboratory are used to select the optimum slurry feeds for the 100-hp and 700-hp facilities. Combustion tests were made with blends of low-sulfur SRC and Pittsburgh seam coal to determine the combustion characteristics and emissions of blends of low-sulfur fuel and non-compliance coals that would permit the burning of the blends in industrial and utility boilers without the need for flue gas desulfurization. The blend burned well but the modified-water-cooled burners developed for SRC firing were required. A blend of char (0.1 percent S) made from wood wastes and Pittsburgh seam coal performed well and could be fired with a standard burner. It was found that the  $\text{NO}_x$  emissions from the high-nitrogen SRC could be effectively controlled by bias firing and staged combustion.

**PLANS FOR THE COMING YEAR** – Construction of the 700-hp oil-fired watertube boiler will be completed. The boiler will be fired with coal-oil slurry mixtures; variables studied will be different coal ranks, coal sizes from utility to micronized, concentrations to 50 percent, residual oils of different chemical compositions, and stabilizers. It will be operated for relatively long periods of time at a given coal size and concentration. The smaller 100-hp boiler will support the larger unit, and a greater number of variables will be investigated in relatively short tests. It will also be used to screen the operability of various burner designs with oil and coal-oil mixtures and for relatively short tests with liquid coal-derived fuels and shale oil. A coal oil slurry test loop will be assembled to study the performance, erosion, corrosion, and fouling in such components as valves, slurry pumps, flow meters, and burner nozzles. Various commercially-available additives for improving slurry stability and handling properties will be evaluated in the Rheology Laboratory and promising ones tested in both the 100-hp and 700-hp boilers.

The handling, pulverization, and combustion characteristics of char from the 75-t/d synthane plant and chars from other synthetic fuel processes will be studied in the 500-lb/hr pulverized solid fuel combustion facility. Alkali sorbents such as  $\text{NaHCO}_3$ ,  $\text{Na}_2\text{CO}_3$ , lime, nahcolite, and trona will be injected into the flue gas at various temperature locations in the solid fuel combustor to study their effectiveness in controlling  $\text{SO}_2$  emissions when burning high-sulfur coal. These additives will be studied and evaluated as  $\text{SO}_2$  control agents for use in smaller industrial and utility boilers. The sulfur-laden additives and fly ash will be collected in a baghouse filter.

## COAL-OIL SLURRY COMBUSTION PROGRAM

INTERLAKE, INC.

DOE - \$2,056,900; Interlake, Inc. - \$534,100

8/23/77 - 11/30/80

**OBJECTIVES** – This program is aimed at verification of previous limited pilot testing on the preparation and use of a coal-oil slurry for injection into the tuyeres of a blast furnace on a full-scale long-term basis. Successful results of the program can lead to greatly decreased quantities of No. 6 fuel oil used as a blast furnace injectant, replacing that amount with coal.

**RECENT WORK AND ACCOMPLISHMENTS** – A review of the basic design and operating parameters has been completed together with a flow diagram and general arrangement drawings. Design work has been started on the facility.

**PLANS FOR THE COMING YEAR** – Design work and specification preparation will be completed. Procurement of materials and equipment will be started, and construction work will commence.

## COAL-OIL SLURRY DEMONSTRATION PROJECT

NEW ENGLAND POWER SERVICE COMPANY

DOE - \$8,636,532; NEPSCO - \$7,140,160

9/30/76 - 12/30/79

**OBJECTIVES** – This project is to demonstrate the feasibility of burning a coal-oil slurry mixture in an 80-Mw utility boiler originally designed to burn bituminous coal but currently firing residual oil. Other objectives are to demonstrate the static and dynamic stability of 30 percent coal and 70 percent oil slurry along with the feasibility of producing a slurry fuel in a continuous-blending facility. The effect of adding small quantities of water and surfactants to improve stability will be examined. In addition, the effect on air emissions will be measured and the potential for equipment erosion, boiler slagging and corrosion, and reduced equipment performance will be determined.

**RECENT WORK AND ACCOMPLISHMENTS** – NEPSCO has directly funded additive development work and slurry blending studies at Adelphi University, the University of Massachusetts, and Tufts University. Promising surfactants have been identified that appear to outperform additive emulsions previously evaluated. Medium-shear slurry-blending equipment has been selected consisting of a conventional pitched-blade turbine agitator. Design of the low-pressure air-atomized burner system has been completed and design of the coal pulverizing and slurry preparation facilities is nearing completion. Additional additives are being evaluated at the University of Massachusetts. Installation of piping and burner components commenced in October 1977.

**PLANS FOR THE COMING YEAR** – The installation of 12 Forney Verloop burners is scheduled for February 1978 with baseline testing on No. 6 oil scheduled in April 1978. The installation of the slurry preparation facility will begin in April 1978, with completion expected in August 1978. Initial slurry preparation and feasibility test firing is scheduled for September 1978.

## COAL-OIL MIXTURE: COMBUSTION IN AN INDUSTRIAL BOILER

ACUREX CORPORATION  
DOE - \$1,684,500; Acurex - \$297,700  
3/77 - 2/80

**OBJECTIVES** – Mixing finely powdered coal with oil offers a simple and potentially economical way to reduce oil consumption in boilers and furnaces. Acurex Corporation is conducting a demonstration program of coal-oil mixture (COM) combustion in an existing package industrial boiler at a tobacco leaf processing plant in Danville, Virginia. Objectives of the program are to design, install, and demonstrate for 1 year a system for COM preparation, combustion, and air pollution cleanup.

**RECENT WORK AND ACCOMPLISHMENTS** – Preliminary support tests on coal grinding and COM combustion were carried out to determine the feasibility of using a wet ball mill to simultaneously grind coal and mix it with oil. Wet grinding tests were conducted at Colorado School of Mines Research Institute using a small research ball mill. These tests substantiated the feasibility of wet grinding, and subsequently a subcontract was awarded to Kennedy Van Saun Corporation to design and build a full scale 12.5-ft-by-6-ft mill. Combustion tests were conducted at Acurex in the Acurex/EPA multifuel furnace test facility. The emission trends of the fuels to be used at the demonstration site were evaluated and combustion performance of COM was compared to that of coal and oil burned separately. The tests showed that COM can be atomized and combusted successfully and produces a flame quite similar to an oil flame. Pollutant trends were, as expected, primarily dependent on the coal fraction; for example,  $\text{NO}_x$  levels ranged from 300 ppm (for straight oil) to 700 ppm (for 50 percent COM). The increased  $\text{NO}_x$  results from the greater fuel  $\text{NO}_x$  in the coal. No major problems were encountered in handling, pumping, or storing COM.

A preliminary design for a complete COM system has been developed. The system consists of: coal conveying equipment and a coal storage silo; a preparation building that houses the ball mill and all pumps and heaters; COM storage and process control tanks; a new retrofit burner that can burn either COM or No. 6 oil; and air pollution cleanup equipment consisting of a baghouse, fan, and stack for flyash collection and a flyash conveying and storage system.

**PLANS FOR THE COMING YEAR** – The final design for the COM system will be completed. The new multifuel burner will be installed during the plant slow period in late spring. The new burner will be checked out, and firing will begin on No. 6 oil. While the new burner is being installed, construction on coal handling, fuel preparation, and fuel storage facilities will begin. Construction and checkout of these facilities will be complete by late fall, and firing on COM will be started.

## DEVELOPMENT AND EVALUATION OF HIGHLY LOADED COAL SLURRIES

ATLANTIC RESEARCH CORPORATION  
DOE - \$155,000  
9/15/77 - Continuing

**OBJECTIVES** – The feasibility of coal slurry fuels containing at least 70 percent coal by weight is being determined. Phase I (in progress) is developing and optimizing stable slurries by control of rheology through composition variables. Phase II will evaluate candidate slurries by means of combustion tests.

**RECENT WORK AND ACCOMPLISHMENTS** – A literature review pertaining to coal slurry fuel technology has been prepared, and Phase I has been started recently. Initial work is directed toward evaluation of gelled carriers composed of either 10 or 30 percent water by weight of slurry, the remainder being No. 6 fuel oil.

**PLANS FOR THE COMING YEAR** – Carrier rheology will be evaluated to determine the cost effectiveness of gellants for water and for oil-in-water emulsions, and candidate carrier compositions will be selected. The particle-size distributions of three different coals (with an emphasis on low-sulfur or beneficiated samples) will be determined for maximum slurry stability and minimum viscosity at 70 percent loadings with each of the candidate carriers.

### POWDERED COAL-IN-OIL MIXTURES PROGRAM

GENERAL MOTORS CORPORATION  
DOE - \$190,000; GM et al. - \$1,000,000  
3/4/76 - 8/15/77

**OBJECTIVES** – This program is to demonstrate the potential in a plant-size environment for using an emulsified fuel consisting of a coal-oil mixture (COM) in an oil-fired boiler without requiring extensive modification or large capital expenditure for additional equipment. Several specific tasks were outlined for this project involving fuel oil-coal selection, pulverizing characteristics, fuel blending, fuel characterization tests, comparative 1-hr combustion tests, and boiler test.

**RECENT WORK AND ACCOMPLISHMENTS** – The overall result of this powdered COM program is that a sufficiently stable coal-oil mixture can be prepared continuously and successfully fired up to 75 percent of maximum boiler rating of an existing (120,000 pph) gas-designed industrial boiler. Many problems were encountered in the slurry preparation plant and most were solved. It is believed that with some preliminary small-scale testing, these problems can be eliminated by proper design of future preparation plants.

The air-atomized burners and the boiler operated well on COM. The coal carbon burnout was about 98 percent. There was no excessive buildup of ash or slagging on the boiler tubes or on the furnace floor, and no evidence of erosion of boiler tubes. Measurements of stack emissions showed that they could be treated by standard technology. There was no major deterioration of boiler efficiency. The boiler steam output was unstable and sometimes did not follow the indicated fuel consumption rate. The boiler tripped off frequently. This behavior was traced to fuel pressure. The cause of the pressure variations could not be completely determined, but variations in viscosity of the coal-oil mixture and transient flow constrictions are most likely candidates.

Based on study data and including installation of the necessary emission control devices, an economic analysis showed the COM concept to be marginally attractive depending upon local coal and oil costs. A final report was prepared and submitted to DOE.

**PLANS FOR THE COMING YEAR** – No additional Government-supported COM work is planned.

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**FUEL EXTENSION BY DISPERSION OF CLEAN COAL IN OIL**  
**GULF WESTERN ADVANCED DEVELOPMENT AND ENGINEERING CENTER**  
DOE - \$129,267  
9/29/77 - 10/29/78

**OBJECTIVES** — This program is designed to demonstrate on a laboratory scale the feasibility of developing a process that cleans coal, simultaneously molecular grafts onto it an oil-compatible polymer coating, and disperses it in oil to produce a temperature-stable liquid fuel. This work represents Phase I of a four-phase program whose ultimate objective is development of a complete production system. It is anticipated that the molecular graft (MG) process will overcome the problem of settling during transportation and storage that has characterized previous attempts to suspend pulverized coal in oil with the aid of surfactants.

The new fuel may contain up to 60 percent coal extender but exhibit few or none of the disadvantages associated with the solid fuel. The dispersed MG-treated coal should be freer from ash and sulfur than coal obtainable by other commercially feasible preparation methods because the MG technique permits finer grinding to release more impurities. The accompanying disadvantage of increased water retention is eliminated, because the water-repellent coating allows substantial moisture removal by physical means. Processing could be carried out at the minehead, allowing pipeline transmission of the finished product. Because the chemical add-on imparts a permanent surfactant effect to the coal particles, settling caused by gravity or temperature effects should be minimized. The resulting fuel should be compatible with standard pumping and burner equipment, with few of the clogging or nonuniform feed problems encountered with other coal slurries.

**RECENT WORK AND ACCOMPLISHMENTS** — All work performed has been of a preliminary nature. Laboratory equipment is being procured and/or adapted for setup at the laboratories of the Natural Resources Group (operated by New Jersey Zinc Company) at Palmerton, Pennsylvania. Also being procured are process reactant materials: coal, benzene, monomer, grafting initiator, and catalyst (from the library of GW proprietary materials).

**PLANS FOR THE COMING YEAR** — A complete laboratory process will be designed and set up to crush, MG/react, clean, dewater, grind, and disperse coal in No. 6 fuel oil. Investigation of process variables will include hydrocrushing, sink-and-float cleaning, hydropulverizing, froth flotation cleaning, dewatering, dispersing, and size reduction techniques. Laboratory experiments will be carried out on molecular grafting coal particles, using a variety of low-cost monomers, grafting initiators, and catalysts. At least 50 candidate molecular-grafting formulas will be investigated, and their dispersion stabilizing ability determined. Other studies will concern fluid maintenance, solution makeup, treated particle dewatering characteristics, MG organophilic and hydrophobic characteristics, viscosity control, shelf and pot life, and low- and high-temperature stability. Both reacted and unreacted coal will be compared for sink-and-float and froth separation efficiency to determine optimum particle size, floating agent requirements, and level of cleaning. Measurements will be made with several particle sizes for both surface and absorbed moisture using centrifugal, cyclone, and ultrasonic centrifuge dryers. Optimum reactants, procedures, and process variables will be selected. They will be integrated into a system design of a laboratory-scale production model that will produce sufficient dispersed coal-in-oil slurry for testing. Plans will be developed for Phase II of the four-phase program for developing an economically feasible process to produce coal-in-oil mixtures.



## CATALYTICALLY SUPPORTED THERMAL COMBUSTION OF COAL-DERIVED LOW-BTU GAS

ENGELHARD MINERALS & CHEMICALS CORPORATION

DOE - \$243,095  
7/29/77 - 10/30/78

**OBJECTIVES** – Catalytically supported thermal combustion will be demonstrated as a means of accomplishing high-efficiency essentially emissionfree combustion of low-Btu gases obtained from coal gasification. Catalyst system designs will be evaluated at simulated gas-turbine operating conditions to demonstrate that a range of low-Btu coal gas fuels can be combusted with low emissions and high-combustion efficiency with acceptable pressure losses. Design information and operating range will be defined.

**RECENT WORK AND ACCOMPLISHMENTS** – Engelhard's high-temperature reactor Unit-4 has been modified to allow separate preheat and injection of low-Btu coal gas blends, air, and steam into the reactor. The reactor includes a flashback sensing zone and a catalytic combustor sector. Precommissioning tests have been initiated.

**PLANS FOR THE COMING YEAR** – Work will include mapping the region of operability of test reactor Unit-4 without flashback in preheated low-Btu gas fuel/air mixtures, selecting an optimum catalyst/catalyst configuration for low-pressure losses and high-combustion efficiency, and characterization performance testing of this selected configuration. A limited study to determine the potential for catalytic cracking of ammonia to nitrogen and hydrogen will be carried out.

## POWDERED COAL AS A CLEAN ENERGY SOURCE

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$75,000  
1977 - Continuing

**OBJECTIVES** – This project is aimed at developing powdered coal (particle size less than 20 microns) as a clean alternate or additive fuel to natural gas or oil for use in furnaces, boilers, turbines, and diesel engines.

**RECENT WORK AND ACCOMPLISHMENTS** – The preparation, characterization, beneficiation, grinding energy requirements, and diesel engine and direct-fired gas turbine burning characteristics of Powdered Coal-Oil Blends (PCOB) are the major thrusts of this new project. Major accomplishments during the first year were: preparation of eleven 55-gal drums of PCOB with 2 micron average particle-size coal; preliminary evaluation of pumping and coal burning characteristics of PCOB in a diesel engine, with encouraging results; initiation of a Special Research Support Agreement (SRSA) between PERC and the University of Utah to determine PCOB grinding energy requirements; initiation of an Interagency Agreement between PERC and the Naval Ordnance Station, Indian Head, Maryland, to carry out magnetic, centrifugal, and dense media gravimetric separation of pyrite and heavy minerals from PCOB, and completion of a SRSA between PERC and the University of Pittsburgh to evaluate the use of powdered coal as a natural gas substitute for sewage sludge incineration.

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**PLANS FOR THE COMING YEAR** – Work for the coming year will emphasize the characterization of PCOB as an alternate fuel for stationary medium-speed diesel and direct-fired gas-turbine cogeneration applications. Work will continue, as required, on PCOB preparation and characterization in terms of particle-size distribution, viscosity, and the like. The environmental problems associated with PCOB use will be identified, and applicability of available control technologies assessed.

### **COAL GRINDING STUDIES**

**KENNEDY VAN SAUN CORPORATION**  
DOE - \$839,795  
9/29/76 - 9/19/78

**OBJECTIVES** – The various coal conversion processes under investigation require a certain amount of feedstock preparation, the minimum being size reduction of the coal to an optimum size consist. Achieving an advantageous size consist can have a positive impact upon the technical and economic aspects of the conversion process. The objective of this coal grinding research contract is to demonstrate how to minimize the amount of undesirable fine material produced while maximizing the production rate per unit of energy when grinding a particular coal of known size consist to a desired size consist.

**RECENT WORK AND ACCOMPLISHMENTS** – Construction of the testing facility housing a dry-ball mill system, a ball-and-race mill system, a hammer mill system, and a wet-ball mill system has been completed. Five representative coal samples—Anthracite coal, washed Lower Freeport bituminous coal, washed Western Kentucky No. 9 bituminous coal, raw Ohio No. 9 coal, and raw Wyoming subbituminous coal—have been delivered to the testing site. Laboratory analysis of their comminution behavior in a ball mill and a ball-and-race mill has been completed. Math models are being prepared under subcontract arrangement with the Penn State University. Grinding tests are underway.

**PLANS FOR THE COMING YEAR** – Testing of the coals will be completed and the findings published as a DOE Coal Grinding Handbook.

### **FLY ASH REMOVAL BY ELECTROSTATIC PRECIPITATION**

**GRAND FORKS ENERGY RESEARCH CENTER**  
DOE - \$330,000  
10/1/76 - Continuing

**OBJECTIVES** – This work is attempting to develop a reliable basis for designing and sizing Electrostatic Precipitators (ESPs) for high-resistivity Western coals, based on data that can be obtained from laboratory procedures performed on relatively small samples of coal derived from core drilling. New methods of fly ash conditioning will also be investigated. No satisfactory basis now exists for sizing ESPs for high-resistivity fly ash, other than the practice of a wide margin of overdesign, which has resulted in unnecessary cost and inadequate control. Another objective is to determine, characterize, and develop control measures for fine particulate generated during the combustion of lignite and Western subbituminous coals. These coals contain atomically dispersed alkali cations that can react with the coal sulfur during combustion to form very fine sulfate particulate in the fly ash.

Studies will also be performed on sulfates generated as the result of dry injection of alkali into stack gases as a second-generation technique of flue gas desulfurization for low-sulfur coals.

**RECENT WORK AND ACCOMPLISHMENTS** – Construction of a new pilot-scale ESP was completed and shakedown tests were performed on it and the furnace. A problem with carbon burnout in the controlled-turbulence combustor was solved by adjusting the proportions of primary, secondary, and tertiary air. Plans for testing of nahcolite and trona (naturally occurring forms of sodium bicarbonate) for ESP conditioning and flue gas desulfurization have progressed in cooperation with Utah Power and Light (UP&L), including baseline testing at the Naughton Power Plant on a high-resistivity fly ash from Kemmerer, Wyoming, subbituminous coal. Laboratory studies were performed on the dielectric properties of fly ash to complement extensive work on resistivity. Changes in dielectric constant along with frequency, temperature, humidity, and ash composition are needed to help explain charge transfer and electrode polarization effects in an ESP. Comparison of the dielectric behavior of fly ash with that of compositionally similar materials permits analyses using electrical theory not previously applied to ESP.

**PLANS FOR THE COMING YEAR** – The tests on dry injection of nahcolite for  $\text{SO}_2$  control and ESP conditioning will be completed. If successful, field tests will be conducted in cooperation with UP&L. ESP efficiency data will be obtained on the pilot unit for a complete range of operating variables on three standard Western coals spanning the range of high- to low-resistivity. Corresponding field data will be obtained on the same coals to relate the laboratory results to commercial design and operating parameters. These data will then be used as points of reference in ranking studies on a large number of additional coals. Laboratory studies on dielectric constant and resistivity will be continued with emphasis on absorption of moisture and other gases on the fly ash surface. A new instrument for differential thermal analyses and thermal gravimetric analyses will be applied. Mathematical modeling of ESP performance will be extended and applied to data from the pilot plant and the field. Fine particulate will be studied using the scanning and analyzing electron microscope to determine the distribution of elemental composition with size.

### **SULFUR EMISSION CONTROL FOR LIGNITE AND SUBBITUMINOUS COAL COMBUSTION**

**GRAND FORKS ENERGY RESEARCH CENTER  
DOE - \$500,000  
10/1/76 - Continuing**

**OBJECTIVES** – This work involves development of alternative methods for control of  $\text{SO}_x$  emissions from the burning of low-sulfur lignite and Western subbituminous coals, and further, to reduce cost, improve reliability, increase removal efficiencies, and improve the characteristics of wastes to alleviate disposal problems. Although most Western coals are low in sulfur content, few

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been achieved in the laboratory under favorable conditions of low sulfur content and high alkalinity. In addition, laboratory tests are conducted on the physical properties and solution chemistry of fly ash and scrubber sludge to assess potential disposal problems. Testing on the 5000-acf/m scrubber was successfully completed, and a commercial scrubber was constructed by the Square Butte Electric Cooperative for a new 450-Mw cyclone boiler fired on North Dakota lignite.  $\text{SO}_x$  removals of up to 85 percent were attained using only fly ash at a typical coal sulfur content of 0.8 percent; under worst conditions of 1.3-percent coal sulfur content, supplementary lime was required to reduce emissions to FNSPS levels. Utilization of the alkali in the fly ash was shown to be increased by reducing the pH or by increasing the concentrations of soluble sodium or magnesium in the recycle scrubber liquor. The test scrubber remained essentially free of scale throughout the 8-month test program. Tests completed on the 130-scf/m laboratory scrubber confirmed that high concentrations of sodium and magnesium improved fly ash alkali utilization and increased  $\text{SO}_x$  removal efficiency. In addition, control tests were performed on a North Dakota lignite as a standard of comparison for testing on other fly ash materials. In the laboratory, methods of calibration were developed for a new analytical instrument, the inductively coupled argon plasma (ICAP) spectrometer. This instrument is being used to analyze scrubber liquor and sludge leachate for up to 38 major and trace elements; it was specifically used to perform a detailed elemental material balance on the 5000-acf/m scrubber. Tests in a 6-inch atmospheric fluidized-bed combustor were completed on ten coals representing the full range of variation in ash analyses for lignites and Western subbituminous coals. During fluidized-bed combustion the alkali in these coals captures about 50 percent of the coal sulfur without recycling of ash; sulfur capture increases to 60 percent when fly ash from the primary cyclone is recycled back to the bed.

**PLANS FOR THE COMING YEAR** – The 130-scf/m scrubber will be used to survey  $\text{SO}_2$  removal efficiencies and scaling rates for a wider variety of fly ashes. Injection of nahcolite (a naturally occurring form of sodium bicarbonate) into flue gas for  $\text{SO}_2$  control will be investigated, and if successful, field tests will be conducted in cooperation with Utah Power & Light. Dry sorbents are expected to find special application in the semi-arid regions of the West. Treated and untreated scrubber sludge and spent dry sorbents will be leached with distilled water and solutions representing ground waters to determine solubilities of elements under simulated disposal conditions. Work on FBC of lignite will be continued and expanded under funding as a separate project. Plans for FBC using lignite call for preliminary scaleup under contract and more detailed optimization in a new 18-inch AFBC to be built at GFERC.

## PARTICULATE ANALYSIS INSTRUMENTATION

LEEDS & NORTHRUP COMPANY

DOE - \$106,095

5/4/76 - 3/30/78

**OBJECTIVES** – Effective particulate control is required for successful development and eventual commercial implementation of advanced combustion and coal gasification systems. Particles entrained in the combustion off-gases must be removed by cyclone and filtering processes. In the case of direct combustion gas turbine systems, residual particle loadings are known to be a major cause of turbine blade erosion. The objectives of this project are to develop an on-line instrument for measuring particulate loadings and particle size in high-temperature pressurized gas streams and to evaluate the resultant instrumentation on pressurized fluidized-bed combustion systems.

**RECENT WORK AND ACCOMPLISHMENTS** – An on-line particulate analysis instrument has been developed for monitoring and analyzing the concentration and size of particles in the gas cleanup stage of advanced combustion systems. It uses optical scattering from laser-illuminated particles by across-the-duct measurements. A prototype instrument was designed, built, and then evaluated on the fluidized-bed combustor at Argonne National Laboratory (ANL). The piping arrangement at ANL permitted measurement of particles after the secondary cyclone and final filter. The data from the on-line instrument were compared to data taken on extracted samples. The results show good correlation for both concentrations and mean size for loadings of 0.1 to 2 grams/m<sup>3</sup> at standard conditons.

**PLANS FOR THE COMING YEAR** – The prototype instrument is to be delivered to the Curtiss Wright Corporation where it will be installed between the final filter and the turbine inlet of the small gas-turbine fluidized-bed system. The gas duct at that location is a 4-in I.D. refractory-lined spool piece. Loading and size measurements will be obtained at variable distance from the gas stream to determine the uniformity of particles across the duct diameter and to verify its applicability to larger ducts and lower loadings.

## PARTICLE MEASUREMENT IN FBC SYSTEMS

SPECTRON DEVELOPMENT LABORATORIES, INC.

DOE - \$17,219

5/76 - 2/77

**OBJECTIVES** – This work involved an assessment of the capability of an advanced diagnostic technique to make required particle field measurements in FBC systems. Gas turbine operation in coal processing facilities requires turbine blade performance in particle-laden hot gas flows. Efficient removal of particulates from the flows used to drive turbines is imperative. An on-line real-time instrument to monitor and maintain the particle environments is essential equipment in the operation of these facilities. Optical particle measuring techniques represent a viable alternative to mechanical sampling techniques. These techniques do not disturb the flow mechanically and provide near real-time data acquisition.

**RECENT WORK AND ACCOMPLISHMENTS** – A laser light scattering instrument called a Particle Morphokinometer (PM) system was used in this program. Key features of the instrument for the FBC application include in-situ measurement, simultaneous size and velocity measurement, and electronic data processing and management. The PM system was installed and operated for 3 months at Argonne, in the FBC facility to measure particle size and velocity in the combustion gas at several different stations. The system was used in the backscatter mode. Although not optimally designed for the ANL installation, the PM system demonstrated the ability to obtain measurements in high-temperature high-pressure environments and to respond to particles ranging in size from 0.2 to 70 microns.

SDL compiled a report, *Laser Interferometer Analysis of Flue Gas Particles from a Fluidized-Bed Combustor*, describing the series of tests performed at ANL to accomplish the following objectives: demonstrate that laser interferometer measurements could be made through windows exposed to temperatures and pressures present in the fluidized-bed combustion regime; compare laser interferometer measurements with mechanical sample measurements; and demonstrate that

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such instrumentation could be operated efficiently by personnel who had little or no experience with it.

**PLANS FOR THE COMING YEAR** — The contract has been completed.

## **TECHNICAL SUPPORT FOR COAL CONVERSION AND UTILIZATION RD&D PROGRAMS**

MITRE CORPORATION  
DOE - \$4,690,087  
6/76 - 6/78

**OBJECTIVES** — A broad range of program planning, systems engineering, and systems analyses is being provided for the Coal Conversion and Utilization RD&D programs within the Division of Power Systems and the Division of Coal Conversion. These tasks are intended to provide the individual program managers with timely planning, implementation, and evaluation data on the RD&D programs for direct combustion, advanced power conversion, and coal conversion systems.

**RECENT WORK AND ACCOMPLISHMENTS** — Assistance to the Direct Combustion Program consisted principally of: providing on-site review and engineering for the startup and initial operation of the 30-Mw atmospheric fluidized-bed pilot plant at Rivesville, W. Va.; reviewing specifications and recommending changes for the steam generator, control system, and materials handling equipment for the Morgantown (MERC) Component Test and Integration Unit (CTIU); preparing a mathematical model and digital simulation of the MERC CTIU control system; and reviewing specifications and drawings as they relate to the DOE involvement in the International Energy Agency's (IEA) 20-Mw pressurized fluidized-bed combustion (PFBC) boiler pilot plant at Grimethorpe, England. Systems analyses under the Direct Combustion Program included a state-of-technology assessment for hot-gas cleaning systems and the development of a dynamic model of fluidized-bed combustors to facilitate design of both the combustor and its control system. Environmental analyses were performed for the Anthracite Culm and Coal-Oil Mixture (COM) Programs. Environmental assessments were prepared for the COM Program and the Rivesville and Morgantown CTIU facilities.

In the area of technology exchange, the Fluidized-Bed Combustion Technology Exchange Workshop, the Coal-Oil Mixture Combustion Technology Exchange Workshop, the Fuel Switching Forum, and the Fifth International Conference on Fluidized-Bed Combustion were organized, and the proceedings published. Systems analysis and systems engineering for the three COM demonstration projects (New England Power Service Company, Acurex-Aerotherm, and Interlake) as well as the COM project at PERC consisted of design evaluation, review of reports and proposals, development of test plans, and specification/review of instrumentation and measurement requirements. In addition, the technical and economic aspects of ultrafine coal were analyzed. Systems engineering tasks for the Advanced Power Conversion System (APCS) Program included: thermodynamic analysis of power cycles; designing of test fixtures; preparation of test plans; review and evaluation of contractor-prepared reports; system integration and analysis; and preparation of updated project descriptions. Analytical efforts were directed principally toward heat and mass balances of the integrated gasifier/combined-cycle designs of High-Temperature Turbine Technology (HTTT) contractors and evaluation of coal-fired gas-turbine power cycles using steam injection. The HTTT Project was further assisted by organizing and coordinating the efforts of the Phase I Evaluation Panel; systematically comparing performance and development plans for the turbine

subsystems of the HTTT contractors; and comprehensively reviewing the engineering aspects of Phase I results (including an assessment of commercialization prospects and the implications of test vehicle scale).

In support of the Ceramics Technology Readiness Project, a systems integration effort is underway to assure that the ceramic materials screening tests scheduled to be carried out at MERC in 1978 will be timely, comprehensive, and significant as a measure of a material's worth in a high temperature, coal-fueled gas-turbine environment. This effort includes designing and overseeing the construction of the high-temperature ( $> 2500^{\circ}\text{F}$ ) test section. An on-site assessment of European technology and experience related to coal-fired primary heaters for closed-cycle gas-turbine power systems was prepared in support of the Advanced Combustion Heater Project.

Environmental analyses were prepared for the refineries selected for demonstrating use of coal-derived synthetic crude oil (PON FE-8) and for the low-Btu gasification demonstrations at small industrial/institutional facilities (PON FE-4). An environmental impact statement was prepared for the University of Minnesota site in the low-Btu project. For the Direct Combustion and APCS Programs, detailed program description documents were prepared as a means for widespread communication of program objectives and content. Major alternative methods for economic evaluation of new utility power system technologies were assessed and illustrated by a reevaluation of the economic performance of the technologies treated by the Energy Conversion Alternatives Study (ECAS). Finally, contributions were made to the DOE Market-Oriented Program Planning System (MOPPS): economic parameters of several alternative fossil energy technologies were determined; MOPPS mission technologies described; and preliminary MOPPS results reviewed.

**PLANS FOR THE COMING YEAR** — Technical review and systems engineering support of RD&D programs will continue to be provided. Program plan description documents will be updated and economic and policy issues will be analyzed in accordance with DOE program needs. The impact of environmental standards (especially water use and the proposed New Source Performance Standards for emissions) on DOE coal RD&D programs will merit special attention.

## **ASSESSMENT OF PROJECT MANAGEMENT PROCEDURES**

**PATHFINDER, INC.**  
DOE - \$49,500  
5/9/77 - 12/9/77

**OBJECTIVES** — The project management procedures assessment is designed to provide program management reviews and in-depth evaluations of major capital projects either contained in the Division of Coal Conversion's program or identified by DCC management staff. These assessments are focused on strategic planning, management reporting systems, cost reporting/control systems, budget/schedule evaluations, contingency planning, multi-contractor coordination, and program feasibility analyses. Specific tasks have been selected by DOE. These include, but are not limited to (1) providing management insight into major project activities and the management-level schedules that are reflections of these activities and (2) reviewing and recommending changes to the project control and reporting procedures used within DCC to coordinate the efforts of DOE, DOE industrial partners, and other project support entities.

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**RECENT WORK AND ACCOMPLISHMENTS** – An in-depth written assessment of existing and proposed project control and reporting documents has been completed. This assessment focused on the practicality of the documents and recommended that DOE prepare a Project Management Plan Guide for utilization by DOE and DOE industrial partners in the preparation of management plans. In addition, a cost estimating classification and contingency policy has been drafted. This policy is intended to be included in the Project Management Plan Guide. Management audit of the atmospheric pressure fluidized bed/combustion/component test and integration unit (CTIU), and project execution strategy reviews for the uranium enrichment program have also been conducted.

**PLANS FOR THE COMING YEAR** – Work will include the completion of the Project Management Plan Guide, assistance in its initial incorporation as a management tool, and continued program management reviews and in-depth assessments of major capital projects as selected by DOE.

### DESIGN AND TECHNICAL SUPPORT

#### OAK RIDGE NATIONAL LABORATORY

DOE - \$320,000

4/1/76 - Continuing

**OBJECTIVES** – This effort will provide design and technical support to DOE/FE over a broad range of engineering assignments.

**RECENT WORK AND ACCOMPLISHMENTS** – Studies and assessments during FY 1977 included a technical/economic assessment of hydrogen production by the steam/molten iron process, assessment of a moving bed system for cleanup of raw gasifier gas, a preliminary technical/economic evaluation of two flash hydrolysis processes, an investigation into the state-of-the-art of hot gas purification processes, and a survey and appraisal of heat recovery processes. Draft reports have been prepared for each of the tasks.

**PLANS FOR THE COMING YEAR** – Following review and comment of the project draft reports, these reports will be issued and new tasks will be undertaken in support of DOE/FE.

### TECHNICAL SUPPORT FOR FOSSIL ENERGY

#### PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$500,000

1977 - Continuing

**OBJECTIVES** – The objective of this project is to provide technical assistance to the Major Facility Project Management (MFP) Division of Fossil Energy by supplying expert advice and consultation services, and by performing experimental evaluations of process equipment to be incorporated into large-scale coal conversion plants. Current efforts are being directed toward the development of a dry coal extrusion feeder. Successful development and implementation of this coal feeding concept would eliminate the design and operational difficulties currently experienced with lock hopper feed systems now used with many high-Btu gasification processes. Extrusion feeders have been employed in commercial polymer applications for several years, but the problems associated with employing these devices to feed dry coal into reactors operating at extreme pressures and temperatures have not been fully explored.



**RECENT WORK AND ACCOMPLISHMENTS** – Preliminary design for the extrusion feeder test facility, which will permit initial hardware evaluation based on tests of dry coal feed into a cold high-pressure receiver, has been completed and procurement of necessary equipment initiated.

**PLANS FOR THE COMING YEAR** – Construction of the extrusion feeder test facility will be completed and shakedown tests into a cold receiver started. Experimental work will be directed toward developing an extrusion feed system with an effluent in the form of a spray or finely divided particles, as required for short residence time gasification processes. Also, testing of novel coal dryers will be initiated. Other tests will be performed in response to demand.

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# *FUEL CELLS*

The fuel-cells activity was transferred from the Conservation Program to the Fossil Energy Program late in FY 1977; therefore, this work will be summarized rather than presented as individual contract writeups. A new energy-conversion alternative, fuel cells are devices that electrochemically convert fuel energy to electricity in a highly efficient and environmentally acceptable manner. Because of their unique features, fuel-cell power plants can be factory built and located close to the point of electricity demand, reducing energy losses associated with transmission and distribution and permitting effective use of waste heat. The modular construction of fuel-cell generators provides a size flexibility from kilowatt to megawatt capacities. The essentially non-polluting, practically noiseless operation permits location in large load centers and even on site in urban, rural, and residential areas. Measured emissions from an experimental fuel-cell power plant were much lower than the Environmental Protection Agency (EPA) standards for modern conventional fossil-fueled central station generators. With current technology (phosphoric acid electrolyte), fuel-cell power plants can utilize methane, synthetic natural gas, propane, ethanol, methanol, light distillates, and hydrogen-rich waste gas streams.

A fuel-cell power plant comprises a fuel processor, power section, and inverter. The fuel process converts a hydrocarbon fuel into a gaseous mixture of hydrogen and carbon dioxide and/or carbon monoxide. If the product gas is to be used in first-generation phosphoric acid fuel cells, the carbon monoxide is reacted with steam recovered from the power section to generate hydrogen and carbon dioxide. If the product gas (such as gasified coal) is to be used in second-generation molten carbonate fuel cells, the carbon monoxide is used directly as fuel by the power section. The Energy Conversion Alternatives Study (ECAS) estimated that a large central station power plant using molten carbonate fuel cells integrated with a coal gasifier could operate at a projected efficiency of 50 percent from coal to AC electricity. The fuel-cell power section consists of many individual cells in which the processed fuel and oxygen from the air are reacted to produce DC electricity. Because the conversion of fuel to electricity is an electrochemical process, the conversion efficiency is very high and is achievable for any plant size and at part load. The last subsystem of the fuel-cell power plant is a static inverter that converts the DC output to AC. The inverter produces a waveform compatible with conventional electrical supplies generated by rotating machinery.

Fuel-cell systems evolving within the next 5 to 10 years appear strongly viable in the following applications: industrial cogeneration where advantage is taken of the high electrical efficiency and compatibility with environmental constraints; integrated energy systems for residential and commercial buildings and district heating including operation from waste- and coal-derived fuels; electric generation for utilities including peaking and intermediate cycling loads, spinning reserve, small dispersed power plants, and baseload from coal. Other applications are under investigation including product petroleum pipeline pumping, gas pipeline compression, and electric hybrid vehicles.

The objective of the DOE Fossil Energy Fuel-Cell Activity is to develop the technology to commercialize fuel-cell power plants for electric utility applications, industrial cogeneration, and building total energy systems in the near term, and to develop advanced, higher efficiency, economically competitive fuel-cell technologies for all end-use applications in the longer term. To achieve this objective, work is divided into four subactivities discussed in the following paragraphs.

*4.8-Mw Electric Utility Power Plant Development.* This subactivity consists of design and fabrication of a 4.8-Mw fuel-cell power plant with subsequent integration of the hardware into a utility grid for testing. The system is being constructed at United Technologies Corporation (UTC). The project is cost-shared by DOE, the Electric Power Research Institute (EPRI), and UTC. Consolidated Edison has been selected as the host utility, and the power plant is to be installed and tested in New York City. The overall objective of the project is to establish the operational feasibility of a fuel-cell system in the technically and economically competitive market that faces all candidate equipment for the generation of electricity. This power plant is a modularly constructed, truck transportable, energy conversion device consisting of fuel processor, power section, power conditioner, and ancillary equipment to maintain appropriate process balance. Because of its modularity, as well as the virtual absence of pollutant generation, it has unlimited siting flexibility. It can serve as a component of central station generation, with particular application for intermediate and peaking loads, and as a dispersed generator for utilities, industries, buildings, and community systems. In the latter mode, the conservation of fuel and dollars is maximized since cogeneration becomes an available option. The operating temperature of the system, 375°F, results in the availability of process steam and low-grade heat for environmental conditioning. The technological feasibility of the phosphoric acid fuel cell for producing electric power has been demonstrated in systems up to 1 Mw in size. The 4.8-Mw module is the basic building block for a power plant that can satisfy the minimum capacity requirements of a utility.

Accomplishments to date include execution of contracts for construction of the 4.8-Mw fuel-cell power plant and the spare parts required for validation and supplemental testing in a host utility. The design and design confirmation phases of the program have been completed and projected performance, meeting specification goals, has been substantiated through subcomponent testing and modeling. Another contractual action, selection of a host for integration of the power plant in a utility grid, has been completed.

*Phosphoric Acid Systems Development.* This subactivity is in the transition phase between development in laboratories and the commercial marketplace. These systems require improved performance, longer life, and an extension of their range of application. Furthermore, additional industrial suppliers of phosphoric acid fuel cells are required to provide a competitive environment. Technology demonstrations (such as the 4.8-Mw power plant previously described and the 40-kw power plant described below) will be useful, but significant improvements will be required. These technology improvements must come in areas of cost reductions, endurance extensions and reliability, and performance improvements. This subactivity, which includes all nonelectric utility applications of phosphoric acid fuel cells, has the objectives of developing fuel-cell systems for specific applications as the last step toward prototype demonstration and providing technology to

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of the availability of waste heat at the application site and the reduction in distribution and transmission losses. The fuel-cell power system is a favorable prime mover for the integrated energy system application because of its modularity, flexibility, and minimum impact to the environment. The integrated energy systems have application in residential and commercial buildings and industrial plants. The power-plant performance specifications are different for those applications; however, the improved fuel utilization efficiency is equally applicable. The primary limitations associated with conventional powered total energy systems in the past have been reliability and siting flexibility. Fuel-cell systems provide an opportunity for overcoming these limitations. Fuel-cell-powered total energy systems would replace natural gas and oil burning systems that provide space and process heat. They would also replace conventional grid-supplied electrical power. These fuel-cell systems could be transitioned to new fuel supplies such as synfuels or coal gasification products. In this area, the power-plant capacity requirements are small relative to a utility application; therefore, potential suppliers are not faced with the prospect of long and costly development and scaleup programs, and users would not be subjected to lengthy and costly field construction. Most of the emphasis to date has been on utility applications thereby leaving the field of on-site applications open to competition. This effort has been structured to provide such competition.

The achievement of significant cost reductions via technology is also receiving major emphasis. Specific efforts involve: improving cell endurance, developing an integral cooler for lower cost and greater durability, developing a design that eliminates the need for nitrogen inert pressurization, improving performance via higher operating temperature and pressure, and developing a new low-cost integral cell stack concept. It is necessary to continually verify that proposed changes for purposes of increased performance or reduced cost do not also reduce stack endurance.

In the OS/IES area, emphasis is on the development of kilowatt-size power plants. Of these, the 40-kw modification and development effort is in the most advanced stage (engineering and development). A 40-kw pilot plant was originally developed under gas utility and United Technologies Corporation sponsorship and was the starting point for this development effort. This pilot power plant has operated for over 10,000 hours. The current effort, cost-shared with the Gas Research Institute, is directed toward cost, reliability, endurance, and performance improvements of the pilot power-plant design to develop a viable, proper functioning 40-kw field test power plant.

*Molten Carbonate Systems Development.* This subactivity is designed to bring about the earliest feasible commercial use of molten-carbonate fuel-cell systems, so that the environmental and resource conservation benefits of these clean, efficient, coal-fueled systems can be realized. Replacement of gas and oil with the minimum amount of coal is facilitated by high system efficiency. Sulfur is removed to low levels from the fuel stream, and because the fuel is oxidized electrochemically rather than by combustion,  $\text{NO}_x$  emissions are very low. The environmental impact of this type of coal-fueled generator is therefore relatively small. The primary application of molten-carbonate fuel-cell generators is for baseload electrical generation, and the program leading to this product is well-defined. With appropriate liquid fuels, such as petroleum or coal-derived hydrocarbons, it would be possible to meet intermediate and peaking demand as well with similar benefits. The path to this application is also well defined, should liquid fuels become available. The operating temperature and flexibility of the fuel cell seems well suited to cogeneration of heat and electricity in a variety of flexibility of the fuel cell seems well suited to cogeneration of heat and electricity in a variety of commercial and industrial situations; but this application has not yet been studied in detail.

In a molten carbonate fuel cell, which would be thermally integrated with a coal gasifier, coal is gasified with steam and air or oxygen to form a fuel gas stream rich in CO and H<sub>2</sub>. The gas stream is purified of particulate and sulfur in the cleanup section, then fed to the fuel cell where it is combined electrochemically with air to form CO<sub>2</sub> and H<sub>2</sub>O. This process produces DC electric power, which is transformed in AC in the inverter and stepped up to transmission line voltage levels. Sensible heat from the gasifier and the fuel-cell outlet gas stream is recovered by raising steam that is used in a steam turbine generator. About two-thirds of the power-plant electric output comes from the fuel cells and one-third from the steam turbine. Where plant sizes smaller than about 500 Mw are needed, a gas turbine can be used instead of the steam system with about 5 percent less plant efficiency. The sensible heat in this system is also at a high enough temperature to be used directly in many industrial processes to replace gas or oil furnaces.

To meet the requirements of an electric baseload system, the following goals were established for the molten-carbonate fuel-cell stacks: life of 40,000 hours to meet low maintenance requirements; current density of 150 A/ft<sup>2</sup> at 0.85 V; and first cost (stack only) of \$60/kw at the current density and endurance design points. Successful achievement of these goals will allow for a system design of 50 percent overall conversion efficiency at a competitive capital cost. Cell performance is rapidly approaching the goal specifications, and may well exceed that level before other goals are met. The present current density at 0.85 V is 100 mA/cm<sup>2</sup>. This specification must be met under system operating conditions of temperature (923°K), pressure (10 atm), CO<sub>2</sub> recycle (currently by burner) and in-cell fuel utilization (currently 85 percent).

*Fuel-Cell Applied Research.* This subactivity encompasses two efforts: technology advancement and advanced concept investigation. Technology advancement efforts perform phenomenological research in areas where fuel-cell development has shown that there are basic problems or technological limitations that adversely affect the cost, efficiency, reliability, and/or lifetime of fuel cells under development. In a few cases, the improved knowhow may also accelerate demonstration and commercialization programs by pointing the way to improvements that can be incorporated without major engineering changes. Technology advancement deals with fundamental physics and chemistry in depth far beyond that of development. For this reason, the most advanced sources of scientific expertise from universities and specialized research organizations are used. Advanced concept investigation efforts consist of exploratory R&D to provide sufficient information to evaluate the development costs, risks, and benefits of new fuel-cell concepts. Emphasis is on concepts with the potential for high national benefits but which cannot be evaluated on a sound basis without obtaining additional laboratory, experimental, and/or engineering data. As many promising concepts as can be accommodated within the budget are explored. After eliminating the less desirable ones, additional research is carried out on the candidate technologies with the greatest likelihood of being attractive to industry. In this manner, information necessary to evaluate in greater depth how promising the technology is for commercialization is provided. The same technological data provide a springboard for industrial product development, or in some cases, for additional Government development work from other sources. The fuel-cell applied research subactivity covers the fuel-cell stack and also the fuel processor work designed to increase the range of alternate fuels that the power-plant can use reliably and efficiently.

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# ***ADVANCED ENVIRONMENTAL CONTROL TECHNOLOGY***

This activity is directed toward developing technology that will minimize the environmental impacts of new and existing fossil fuel utilization systems. These efforts are being transferred from the Environmental Protection Agency (EPA) to the Assistant Secretary for Energy Technology's Fossil Energy program within DOE. Management details of this transfer (e.g., specific projects and overall program management responsibilities and roles) are under negotiation. The goal of this transfer is to integrate all R&D efforts relating to a single balanced national program for environmentally safe utilization of coal as a primary fuel source. A complete system approach to the fossil fuel utilization/environmental impact problems will be pursued, considering the tradeoffs among environmental control options and the interactions between control devices and other power systems components. To achieve this overall goal, several specific objectives have been established: develop advanced technologies for coal cleanup prior to use, develop technologies for gas cleanup during the combustion process, develop environmental control technologies to clean post-combustion gases including second-generation flue gas technologies, and determine cost versus environmental impact tradeoffs for environmental control/power systems combinations.

The EPA program achieved several objectives: publication of manuals and data books on coal cleanup including both sulfur and hazardous trace material removal for a variety of coals; design, construction, and 1-year operation of a Meyers process chemical coal-cleaning test facility; development, operation, and initial evaluation/environmental assessment of a 0.63-Mw fluidized-bed combustion miniplant; construction, operation, and initial data on the chemically active fluidized-bed process for converting high-sulfur oil and coal to clean-burning low-Btu gas; and initiation of efforts on second-generation scrubber technologies and high-temperature/high-pressure particulate control methods. The Department of Energy will continue projects designated for transfer and will initiate advanced R&D efforts in environmental control technology. These activities are briefly described in the following paragraphs.

*Pre-Combustion Control.* Coal cleanup efforts will examine both physical and chemical removal of hazardous trace materials, especially metals, as well as sulfur. Coals will include eastern, midwestern, and western high-sulfur coals. The Meyer's process chemical coal-cleaning test facility will be operated to collect additional data on a variety of coals. Longer term research will be directed toward development of new technologies for removal of organic sulfur such as oxydesulfurization, chlorinalysis, and microwave desulfurization.

*Gas-Stream Cleanup.* Gas-stream cleanup activities are composed of four elements: requirement analysis/tradeoff studies, advanced combustion practices, cleanup technology development, and demonstration facilities. An analysis will be made of the requirements for the cleanup systems and the connecting power systems components; for example, low-Btu gasifier/gas turbine and pressurized fluidized-bed/gas turbine. The advanced practices area includes tradeoff studies between various approaches for emission abatement by modification of the coal-firing practices (e.g., staged combustion), as well as design of new combustion concepts. Cleanup technology development will be directed to evaluating and developing high-temperature high-pressure precipitators, baghouse filters, cyclones and centrifuges, granular beds, and advanced concepts, (electron beams, lasers,

microwaves). Demonstration facilities for evaluation of gas-stream cleanup include the pressurized fluidized-bed at Exxon (0.6-Mw miniplant), IEA Grimethorpe (England), Argonne, Curtiss Wright, and American Electric Power. The atmospheric fluidized beds being tested include the Rivesville 30-Mw pilot plant and the Morgantown Component Test and Integration Unit (CTIU). There are also several low-Btu gas facilities. Hot-gas cleanup efforts will be closely coordinated with materials tolerance activities being conducted in gas-turbine work.

*Flue-Gas Cleanup.* Flue-gas (FG) cleanup activities will examine lime/limestone flue-gas desulfurization (FGD) reliability, and advanced flue-gas cleanup. The effort in the lime/limestone reliability area will be directed toward development of a technology that can be presented in a design manual that catalogs critical design and operational parameters as a function of coal type, lime/limestone composition and chemical processes. Design guidelines will be proven in existing full-scale facilities. The advanced FG cleanup thrusts will be directed toward development of more efficient (smaller, cheaper), less energy consuming systems that remove  $\text{SO}_x$  and  $\text{NO}_x$ , either separately or in a combined fashion, and minimize solid waste. The many regenerative processes that have been proposed will be examined from an economic viewpoint. Applied research to dispose and/or utilize coal-process wastes will be emphasized. Instrumentation capable of measuring all process variables (pH, temperature, pressure, etc.) during operation will be developed and evaluated. Systems studies will be conducted to accelerate/compare and quantify the capacity of a control technique to enter the marketplace.

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# MAGNETOHYDRODYNAMICS

The Magnetohydrodynamics (MHD) Power System Program was established to develop commercial-scale electrical power generating plants using coal as the primary fuel. The *ERDA-NSF-NASA Energy Conversion Alternatives Study (ECAS)* concluded that open-cycle MHD (when combined with bottoming steam systems) offers greater potential for significant improvements in overall power system thermal efficiencies than any other advanced power cycle operated with coal. As demonstrated experimentally, the open-cycle MHD generator also has the capability of operating with a working fluid produced by the combustion of coal without the need for removal of the ash and sulfur content. The basic MHD process can result in plant exhaust emissions that will meet the most stringent air quality standards, and the higher operating efficiency results in lower thermal pollution levels. The MHD Program, directed primarily towards open-cycle systems, is based upon significant supporting science and technology activities including laboratory-scale testing and preliminary engineering studies. Exploratory work is also proceeding on closed-cycle systems, concentrating on heat exchanger and generator problems. Closed-cycle development is not as advanced as the open-cycle concept; therefore, this work is addressing basic physical and systems issues that must be resolved prior to considering the closed cycle as a potential alternative.

## PROGRAM REQUIREMENTS

The development and demonstration program for commercialization of MHD in the 1990's will progress sequentially through three phases: development and testing of MHD core components at up to 50 Mw<sub>t</sub>; design, construction, and operation of a pilot-scale demonstration plant; and finally commercial demonstration. Significant overlap exists among the phases, but each one is focused on specific objectives. Phase 1 builds on results from initial applied research to design, develop, and test system components. The primary emphasis is to establish the technical feasibility of critical components such as the MHD generator and then the economic and environmental potential. A key focus of Phase 1 is the Component Development and Integration Facility (CDIF). Phase 1 will provide realistic subscale testing and integration of key MHD subsystems, specifically the coal combustor, generator, and other hot-gas flow train CDIF components. Phase 2 encompasses the scaleup and total integration of a combined steam cycle-MHD system in a pilot-scale experimental plant. The major milestone is start of construction of the Engineering Test Facility (ETF) in FY 1983. During this phase, design, testing, and refinement should improve performance and endurance and lay the groundwork for design of a commercial-scale demonstration plant. Phase 3 emphasizes design, construction, and operation of a commercial-scale demonstration plant along with coordinated research to optimize MHD system performance.

## STATE OF TECHNOLOGY

Development of MHD began during the late 1950's. There are programs in this country and others, notably in the Soviet Union and Japan, with the basic distinction between the U.S. and foreign programs being the emphasis abroad on the use of natural gas in the Soviet Union and fuel oils in Japan. In the United States, the emphasis is on coal as the primary fuel; it is the handling of the gas stream produced by this fuel, with its attendant sulfur and ash content, that yields the major technical considerations marked in MHD systems development. Progress has been made in understanding basic phenomena, evaluating component performance, establishing a design basis for key components and subsystems, and defining full-scale system requirements. The next critical objective



is a realistic engineering demonstration of an integrated MHD power system. Major recent accomplishments, and near-term program expectations are as follows:

***Major Recent Accomplishments***

- Generator – Successful completion of a scheduled 20-Mw<sub>t</sub> 250-hr endurance test under electrode loading conditions simulating commercial service achieving order-of-magnitude improvement in electrode corrosion/erosion.
- Generator – Successful subsonic generator operation at high-magnetic field (5 tesla) representing largest (30 Mw<sub>t</sub>) high field test ever run.
- Combustor – Slag rejection in excess of 95 percent with carbon conversion of 97 percent achieved in first-stage prototype of two-stage combustor for MHD application.
- ETF Conceptual Design – A series of three initial conceptual design studies has been completed providing the first iterative step in ETF design.
- Environmental – An MHD system has been run with 95 percent sulfur removal from high sulfur coal as well as NO<sub>x</sub> reduction to 20 ppm.
- High-Temperature Air Preheaters – Achieved 800-hr cumulative testing at 2700°F on direct-fired regenerative air preheater element under heat transfer, fluidynamic, and seed/slag conditions simulating MHD service; 100-hr continuous run on complete subscale heater at 3000°F using simulated coal firing with ash carryover into heater matrix.
- Superconducting Magnet – Design principles confirmed for large superconducting magnets by operation of 40-ton 5-tesla magnet at design conditions.

***Near-Term Program Expectations***

- Complete Construction of CDIF – The major focus for the first phase of MHD is the 50-Mw<sub>t</sub> CDIF for which ground was broken at Butte, Montana, on May 16, 1976; this facility is at approximate construction midpoint at the date of this report.
  - Delivery of Ash Injection Combustor for Use with First CDIF Power Train – Achievement of this integrated step will lead to testing in the CDIF; ash is being used in lieu of coal to facilitate early testing and to provide scaleup data from smaller development activities.
  - Delivery of First Experimental 50-Mw Channel to the CDIF – This will be achieved as an integrated step leading to testing in the CDIF; initial generation of power from Test Channel 1A is anticipated in FY 1980.
  - Complete Construction and Startup of Coal-Fired Flow Facility – This will be achieved during FY 1979 to provide a development facility for testing at the 20-Mw<sub>t</sub> level.
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## MHD COMPONENT DEVELOPMENT AND INTEGRATION FACILITY CONSTRUCTION

KAISER ENGINEERS  
DOE - \$21,537,000  
9/28/76 - Continuing

**OBJECTIVES** – The CDIF is a major facility necessary to implement the national MHD power demonstration program. This work is designed so that construction activity at the Butte, Montana site can be completed early in calendar year 1979 to allow the start of MHD component testing in September 1979.

**RECENT WORK AND ACCOMPLISHMENTS** – Major construction work began in April 1977. At the end of 1977, physical progress will be approximately 27 percent accomplished, compared to the 23 percent scheduled. The Operations and Services Building was “closed-in” so that interior work can be performed during the winter months. The Main Experimental Building, adjoining the Operations Building, is nearing “close-in” completion, and interior work is also scheduled for the winter months. The foundation for the Coal Preparation Building was completed although it was not originally scheduled as calendar year 1977 work.

**PLANS FOR THE COMING YEAR** – The majority of the plant construction is scheduled for completion during calendar year 1978. The Coal Preparation Building is scheduled for early calendar year 1979 completion.

## MHD COMPONENT DEVELOPMENT AND INTEGRATION FACILITY DESIGN

THE RALPH M. PARSONS COMPANY  
DOE - \$6,150,000  
1/76 - 2/79

**OBJECTIVES** – A test facility is being designed to conduct performance and endurance tests of key hot-gas flow-train components (combustors, nozzles, channels, diffusers, inverters, and magnets) and other MHD test-train support subsystems (coal and seed processing and feed, high-temperature air preheaters, etc.) for coal-fired MHD systems; and to establish the operational characteristics associated with integrated operation of selected combinations of MHD components and subsystems.

**RECENT WORK AND ACCOMPLISHMENTS** – Final design activities have been moving forward. All support system flow sheets and process and instrument diagrams were completed. Designs of the Component Test Building and the Operations and Services Building were released for construction in the spring of 1977. Final design of the Coal Preparation Building was completed and only requires receipt of equipment supplier data for major components to verify the design. For equipment to be located in the yard, final design activities are still in progress. Construction of the CDIF, being performed by Kaiser Engineers, was approximately 27 percent complete at the end of this report period.

**PLANS FOR THE COMING YEAR** – Final design of all facility equipment will be completed. Review and approval of major CDIF system equipment to be supplied will be concluded. Field inspection and engineering services will be provided to support construction activities. To support test program planning, component checkout and system operating tests will be performed.

## TEST INTEGRATION OPEN-CYCLE MHD CDIF

WESTINGHOUSE ELECTRIC CORPORATION

DOE - \$3,543,000

2/14/77 - 2/14/79

**OBJECTIVES** – The MHD/CDIF Test Integration Project has three primary tasks supporting the overall program goal of developing MHD components for which pilot scale data are required: to assure engineering and design compatibility between the first MHD Test Trains, defined as 1A and 1B, and Test Bay Nos. 1 and 2 of the CDIF Test Facility; to assure safe and efficient operation of experimental MHD component test trains in the CDIF Test Facility; and to provide engineering drawings, analyses, specifications, plans, and procedures necessary to accomplish these purposes. This program involves the “writing down” of the MHD Test Train 1A and 1B design requirements; the development of form, fit, and function interface control documents between the MHD Test Train components and the MHD Test Trains in the CDIF Test Facility; the development and/or integration of document and hardware trees, test plans, test specifications, and the formal baselining and change control of these documents.

**RECENT WORK AND ACCOMPLISHMENTS** – Various CDIF/MHD program baseline documents have been developed that provide a basis for detail design and analysis of Test Trains 1A and 1B required interface equipment, test planning, and change evaluation. Included among them are Safety Analysis Plan; Interface Management Plan; Document Change Control Procedure; Instrument and Control Measurement Requirements for Test Trains 1A and 1B; Primary Cooling Water System, Resistive Load Bank, and Test Simulation Model Design Requirement Documents; Baseline Program Documentation Tree; Test Assembly Support Equipment Functional Flow Diagrams and associated assembly, handling, and transportation equipment lists/requirements; MHD Test Train/CDIF Test Facility Interface Requirements; and Checkout Program Plan for Test Train 1A. Each of these documents has been issued in preliminary or final DOE approved versions and is being used as required by the various participants including the Test Integration Contractor. Two major design tasks were completed on schedule: first, Title I or Preliminary Design and Analysis for the Primary Cooling Water System (PCWS) and Title II or Final Specifications issuance for each of the major items of PCWS components; second, reference design for the resistive load bank that is capable of receiving 3 Mw<sub>e</sub> electrical energy from MHD Test Train 1A and converting this energy into thermal energy that is dissipated by the PCWS. Other significant activities include development of top-level fault events and associated fault trees relevant to the CDIF safety analysis task; issuance of process, instrumentation, protection, and interlock diagrams for Test Trains 1A and 1B; and development/implementation of models for the combustion air compressor that simulates compressor performance and associated piping which are being used to obtain systems frequency response data.

**PLANS FOR THE COMING YEAR** – Based on system-level studies and integration efforts that established the identification of and design requirements for items of Test Support Equipment (TSE) needed to conduct the Test Train 1A test program, final designs will be developed and issued for procurement by the end of 1978 for essentially all items of TSE. The design of various MHD test component related equipment, which includes Building 60 PCWS, resistive load bank, central control room test train I&C equipment, and Buildings 50 and 60 Power and I&C Systems, is scheduled to be completed by August 1978. Other major tasks scheduled for completion include Test Train 1A fault tree and failure mode and effects analyses, Test Train 1A controls analysis that

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will demonstrate via suitable analytical models normal system behavior and establish requirements on the control system and/or operational limits, development of Test Train 1A interface control drawings to assure that the MHD test train assembly is physically and functionally compatible with the CDIF test facility and test support equipment, and issuance of a preliminary Test Train 1A test assembly drawing. This drawing will be used to integrate and control the highest level assembly/installation drawings prepared by the suppliers of the MHD Test Train components.

## RESEARCH IN COAL-BASED MAGNETOHYDRODYNAMICS

### PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$4,232,000

1972 - Continuing

**OBJECTIVES** – The major thrust of this program is to obtain design data relevant to two-stage MHD pressurized combustors. This effort is being carried out in three complementary areas: experimental testing in atmospheric and pressurized combustion facilities, mathematical modeling of two-stage combustors, and design of a 50-Mw two-stage combustor.

**RECENT WORK AND ACCOMPLISHMENTS** – Three different configurations of the first stage of a vertical cyclone gasifier-combustor were designed, fabricated, and tested in the atmospheric combustor test facility. The scroll-type vertical cyclone configuration was found to be superior to both side- and top-entry cyclones. Data on residence time, carbon conversion, heat transfer, and air-fuel ratio were obtained. The 5-Mw pressurized combustion test facility was placed in operation, and a coal dryer was installed to permit testing with high-moisture subbituminous coal. Fourteen tests were conducted with oil, bituminous, and subbituminous coals in a first-stage cyclone gasifier-combustor. Computerized data acquisition has been implemented and software development for data analysis is in progress. A unique multi-phase equilibrium computer program was developed and combined with a gas dynamic code and with literature data on coal particle gasification kinetics. The resulting code, Cyclone Gasification Numerical Simulation (CYGNUS), is used to predict the behavior of engineering scale experiments and assist in scaleup design of larger combustors. The design of the 50-Mw two-stage combustor consists of a vertical slagging gasification stage and a close-coupled horizontal combustion stage. It is designed to operate at a pressure of 6 atm with exit temperature of 4650°F and electrical conductivity of 8.3 mhos/m. Most of the ash in the coal will be removed as slag from the first stage. Electrical isolation (20 kV) of the entire combustor is included in the design. Detailed drawings of the combustor design are in progress. In addition, a study was conducted to compare the energy consumption and costs for sulfur oxides removal in open-cycle MHD with conventional flue gas desulfurization. Based on one possible scheme, the MHD cycle compares favorably with conventional flue gas desulfurization.

**PLANS FOR THE COMING YEAR** – An improved design of the scroll combustor (watertube construction) will be designed, fabricated, and tested in the atmospheric test facility. A scale model of the 50-Mw two-stage combustor design will be fabricated and tested in the pressurized test facility. An instrumented test section will be installed in the pressurized combustor to evaluate plasma properties as a function of combustor operating variables. Detail drawings of the 50-Mw combustor, electrical isolation, slag removal and cooling system will be completed. The CYGNUS mathematical code will be supplemented with data from the atmospheric and pressurized combustor tests.

## SCMS SCALING STUDIES

ARGONNE NATIONAL LABORATORY  
DOE - \$23,000  
3/77 - 9/78

**OBJECTIVES** — The CDIF superconducting magnet should serve as a model coil system, with the design being scalable to future large MHD magnets to be used in the Engineering Test Facility (ETF) and full-size baseload systems. Furthermore, the design must be cost-effective and use the experience gained in the design and construction of other large superconducting magnets such as the U.S. superconducting magnet system (SCMS) discussed under the International Program of this section. This project will carry out the conceptual design studies on CDIF, and provide the extrapolation studies of the U-25 bypass magnet design to the baseload MHD magnets.

**RECENT WORK AND ACCOMPLISHMENTS** — A conceptual design study for the CDIF magnet has been completed. Several of the magnet design characteristics are:

|                         |  |
|-------------------------|--|
| On-axis field (tapered) | Inlet at 5.5 T, peak at 6 T<br>Outlet at 4.5 T         |
| Useful field length     | 3 m  |
| Warm bore (tapered)     | Inlet bore is 86 cm diam<br>Outlet bore is 108 cm diam |
| Total ampere-turns      | $12.7 \times 10^6$                                     |
| Operational current     | 5000 amp   |
| Peak field in winding   | 7.3 T  |
| Stored energy           | 172 Mjoules  |

The design uses layer-wound cylindrical saddles assembled on a cylindrical-bore tube. A micarta coil form similar to the one used for the SCMS coil fabrication is suggested for the saddle coil. A series of outer ring girders forms the containment structure for the outward lateral component of the electromagnetic force. The bore tube and the circumferential banding play only a secondary role in supporting this force component. The CDIF magnet is designed to satisfy these field requirements: a peak field on axis of 6.0 T, a distance of 3.00 m from the entrance field value of 5.5 T to the exit field value of 4.5 T, a field taper from the peak field point to the exit (4.5 T), and a field uniformity to within 2½ percent of the desired field at any given point.

**PLANS FOR THE COMING YEAR** — The conceptual design for the CDIF superconducting magnet will be extended to consideration of a commercial-size baseload magnet. Also, coil fabrication modeling using large conductors will be pursued to examine critically the merits of the design

## MHD GENERATOR DEVELOPMENT

AVCO EVERETT RESEARCH LABORATORY  
DOE - \$21,154,000  
1/1/77 - 9/30/80

**ES** — This R&D program is establishing the engineering base for the design and construction of the first CDIF MHD power train as well as investigating the preferred configuration of coal-fired central-station MHD power channels.

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**RECENT WORK AND ACCOMPLISHMENTS** — A detailed performance comparison was made of a channel with rectangular cross-section and a channel with hexagonal cross-section. The performance of the rectangular cross-section channel was found to be superior. An experimental optimization study of the aspect ratio of the supersonic diffuser, with flow blockage, was carried out and the optimum aspect ratio (L/D) was of the order of 5. Oxidation was found to be the main mechanism for degradation of anodic surfaces. Precious metal-clad (e.g., platinum) copper elements were shown to have the potential for maximum endurance under simulated coal combustion conditions. Cathode voltage nonuniformities were shown to be caused by axial shorting through the slag layer. Rigid current controls were shown to be the best choice for local management of currents and voltages along the length of slagging MHD channels. Various consolidation and control circuits were built and tested to determine their operational characteristics and preferred configuration. A new disk channel loft was selected to optimize channel performance taking into account plasma nonuniformities. Under simulated coal combustion products conditions and in a pure radial flow configuration, an enthalpy extraction ratio of 15 percent was achieved.

A Diagnostic Test System (DTS) was designed, fabricated, acceptance tested, and is ready for delivery. The function of this system is to assist in the development of the PERC 5-Mw coal combustor and to evaluate the combustor performance with respect to those operational parameters influencing channel operation and performance such as static pressure, temperature, spatial conductivity, bulk conductivity, slagging characteristics, and flow duct heat loss. The DTS is compatible with existing equipment of the PERC coal combustor development facility.

The High-Temperature Experimental Preheater Facility (two regenerative refractory-type preheaters with ancillary equipment) was employed to supply the inlet air at 2500° to 2700°F for coal-fired combustion experiments with high-temperature clean air, which is analogous to the combustor preheat environment in an MHD plant. An experimental combustor was configured such that ignition, system stability, combustion temperature, and carbon burnout could be evaluated versus the fuel/air ratio. The fuel used was Pittsburgh Seam coal. The key conclusions resulting from these experiments are that the coaxial coal injector/inlet air configuration is satisfactory for tests to obtain combustion data with no apparent limitations with respect to operating time and no susceptibility to localized partial coal pyrolysis resulting in nozzle plugging; the air heater/combustor combination showed no tendency for acoustic or pressure instability during ignition, operation, and when switching from one air preheater to the other; and comparison with predictions based on total coal carbon fed into the combustor suggests that approximately 93 percent carbon burnout was achieved at 36 msec, corresponding to 95 percent of the fuel heating value, with the last 4 to 6 msec of residence time resulting in minimal gain in fuel utilization. A combustion model has been developed that includes a comprehensive description of heterogeneous char oxidation, coupled with particle pyrolysis.

Two reference channels were designed and readied for testing. Both include precious metal capped anodes. One of them is equipped with 3 piece sidewalls, the other with peg walls. Lofting of CDIF 1-A channel was completed. Preliminary design was initiated. The design of an ash-injection combustor for the CDIF was completed and the design package submitted to DOE.

**PLANS FOR THE COMING YEAR** — A new Mark VI size channel will be lofted, designed, and its construction initiated for investigation of transonic channel behavior. Long-duration tests will be conducted to assess performance of improved anode designs and sidewalls construction. Inverter

## SCMS SCALING STUDIES

ARGONNE NATIONAL LABORATORY

DOE - \$23,000

3/77 - 9/78

**OBJECTIVES** – The CDIF superconducting magnet should serve as a model coil system, with the design being scalable to future large MHD magnets to be used in the Engineering Test Facility (ETF) and full-size baseload systems. Furthermore, the design must be cost-effective and use the experience gained in the design and construction of other large superconducting magnets such as the U.S. superconducting magnet system (SCMS) discussed under the International Program of this section. This project will carry out the conceptual design studies on CDIF, and provide the extrapolation studies of the U-25 bypass magnet design to the baseload MHD magnets.

**RECENT WORK AND ACCOMPLISHMENTS** – A conceptual design study for the CDIF magnet has been completed. Several of the magnet design characteristics are:

|                         |  |
|-------------------------|--|
| On-axis field (tapered) | Inlet at 5.5 T, peak at 6 T<br>Outlet at 4.5 T         |
| Useful field length     | 3 m  |
| Warm bore (tapered)     | Inlet bore is 86 cm diam<br>Outlet bore is 108 cm diam |
| Total ampere-turns      | $12.7 \times 10^6$                                     |
| Operational current     | 5000 amp   |
| Peak field in winding   | 7.3 T  |
| Stored energy           | 172 Mjoules  |

The design uses layer-wound cylindrical saddles assembled on a cylindrical-bore tube. A micarta coil form similar to the one used for the SCMS coil fabrication is suggested for the saddle coil. A series of outer ring girders forms the containment structure for the outward lateral component of the electromagnetic force. The bore tube and the circumferential banding play only a secondary role in supporting this force component. The CDIF magnet is designed to satisfy these field requirements: a peak field on axis of 6.0 T, a distance of 3.00 m from the entrance field value of 5.5 T to the exit field value of 4.5 T, a field taper from the peak field point to the exit (4.5 T), and a field uniformity to within 2½ percent of the desired field at any given point.

**PLANS FOR THE COMING YEAR** – The conceptual design for the CDIF superconducting magnet will be extended to consideration of a commercial-size baseload magnet. Also, coil fabrication modeling using large conductors will be pursued to examine critically the merits of the design features.

## MHD GENERATOR DEVELOPMENT

AVCO EVERETT RESEARCH LABORATORY

DOE - \$21,154,000

1/1/77 - 9/30/80

**OBJECTIVES** – This R&D program is establishing the engineering base for the design and construction of the first CDIF MHD power train as well as investigating the preferred configuration for future coal-fired central-station MHD power channels.

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**RECENT WORK AND ACCOMPLISHMENTS** — A detailed performance comparison was made of a channel with rectangular cross-section and a channel with hexagonal cross-section. The performance of the rectangular cross-section channel was found to be superior. An experimental optimization study of the aspect ratio of the supersonic diffuser, with flow blockage, was carried out and the optimum aspect ratio (L/D) was of the order of 5. Oxidation was found to be the main mechanism for degradation of anodic surfaces. Precious metal-clad (e.g., platinum) copper elements were shown to have the potential for maximum endurance under simulated coal combustion conditions. Cathode voltage nonuniformities were shown to be caused by axial shorting through the slag layer. Rigid current controls were shown to be the best choice for local management of currents and voltages along the length of slagging MHD channels. Various consolidation and control circuits were built and tested to determine their operational characteristics and preferred configuration. A new disk channel loft was selected to optimize channel performance taking into account plasma nonuniformities. Under simulated coal combustion products conditions and in a pure radial flow configuration, an enthalpy extraction ratio of 15 percent was achieved.

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**PLANS FOR THE COMING YEAR** — A new Mark VI size channel will be lofted, designed, and its construction initiated for investigation of transonic channel behavior. Long-duration tests will be conducted to assess performance of improved anode designs and sidewalls construction. Inverter



loading, both diagonal and Faraday with nondissipative current control elements will be carried out. The combustion tests employing the refractory regenerative heaters will be continued with Montana Rosebud coal as the fuel. Modeling work will continue with emphasis on the effect of vitiation-heated oxidizer on coal combustion and exploration of optimum particle size distribution versus grinding costs, combustion efficiency, and combustor heat loss. Also, the model will be developed further to describe nonequilibrium mineral matter thermal and state behavior.

## OPEN-CYCLE MHD: CONCEPTUAL DESIGN OF ETF

GENERAL ELECTRIC COMPANY

DOE - \$2,063,000

1/4/77 - 3/4/78

**OBJECTIVES** – This program is developing a conceptual design and capital cost estimate for an engineering test facility (ETF) in which integrated system testing of an MHD/steam combined-cycle power plant can be conducted. Alternative designs of key MHD components are to be evaluated and the preferred designs integrated into a reference design that may form the basis for further design development. A 250 Mw<sub>t</sub> facility size has been chosen as the minimum size at which the component designs are scalable to utility-size components.

**RECENT WORK AND ACCOMPLISHMENTS** – Design requirements and criteria for the ETF have been prepared. An overall system configuration has been developed and trial heat balances produced. Conceptual design for the key components is nearing completion. These key components are the high-temperature air heaters, which are to deliver a preheated oxidizer to the combustor at a minimum of 2700°F; combustor, which is to deliver seeded plasma to the MHD generator at 4600°F, minimum; MHD generator, which must operate with minimum system heat loss and extract approximately 15 percent of the enthalpy in the entering plasma; superconducting magnet, which must produce a magnetic field of up to 6 tesla in the MHD generator; diffuser, which must decelerate the gas exiting the MHD generator and provide pressure recovery by conversion of kinetic energy; radiant boiler, which must transfer enthalpy from the gas to boiler feedwater while providing a temperature versus time profile that allows the thermal decomposition of NO<sub>x</sub> formed in the combustion process; seed condenser, which removes seed (K<sub>2</sub>SO<sub>4</sub>) from the gas; and the steam generating components, which provide steam to the steam turbine generator bottoming plant.

**PLANS FOR THE COMING YEAR** – The conceptual design of the key components will be completed, and recommendations for a preferred ETF configuration developed. Designs for the balance of plant and the overall facility will be prepared. Capital cost estimates and operating cost estimates for the ETF will also be prepared. Iterations on the conceptual design of certain key components will be made, and system performance analysis will be updated. A final report, containing the results of the system study and analysis work, and a reference design for the ETF will be produced.

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## MHD HIGH-PERFORMANCE DEMONSTRATION EXPERIMENT

U.S. AIR FORCE  
ARNOLD ENGINEERING DEVELOPMENT CENTER  
DOE - \$7,214,000  
11/73 - 4/80

**OBJECTIVES** – The goals of this program are to design and fabricate a new 6-tesla magnet and an MHD generator channel, install the magnet and channel in the existing facility at AEDC, and operate the facility to provide experimental evidence that a commercial-sized MHD generator can operate at an isentropic efficiency of 60 to 70 percent and convert 20 percent of the available thermal energy into useable electrical power. This experiment is an important current effort within the DOE MHD program directed toward the achievement of high-generator performance simultaneously with the simulation of commercial-sized MHD generator operating conditions.

**RECENT WORK AND ACCOMPLISHMENTS** – The major effort this year was the assembly of the magnet and channel and the initiation of the burner checkout operation. The burner system consists of an igniter that starts a larger pilot burner, and then the main burner itself. Initially, a series of igniter and pilot runs were performed to ascertain the system response times and to establish ignition repeatability. The burner was successfully fired, first in the manual mode and then with complete automatic operation. During burner operation, two nozzle blocks were damaged beyond repair. The testing was suspended until 1978 when replacement blocks will be available and the planned diagnostics can be utilized. The MHD generator channel copper electrodes, graphite caps, pegboards, and external structure were fabricated by outside contractors and delivered to AEDC. The assembly of the channel was initiated, and the assembly of Section IV was completed. The 18 double-layered, 40-turn pancakes, which constitute the magnet coils, were fabricated by an outside contractor and delivered to AEDC. The force containment structure machining was completed, a trial assembly was accomplished, and the structure was disassembled and shipped to AEDC. The assembly of the magnet was initiated and by the end of September 1977, three of the nine lower pancakes were in place.

**PLANS FOR THE COMING YEAR** – The five channel sections will be assembled; the magnet coils, force containment structure, and insulation will be installed; and the burner calibration tests will be completed.

## MHD COAL-FIRED POWER-GENERATION TEST FACILITY

UNIVERSITY OF TENNESSEE SPACE INSTITUTE  
DOE - \$12,403,792  
12/31/74 - 9/30/78

**OBJECTIVES** – The UTSI facility, simulates the various components of the MHD/steam power plant and of the new DOE test facility being constructed nearby under this contract, so that their performance under direct coal-firing will be better understood. Another major task involves the design and construction of a new facility to be operated largely on eastern bituminous coal that will test the concepts crucial to commercialization of the MHD/steam power plant on a larger scale and for longer periods of time.

**RECENT WORK AND ACCOMPLISHMENTS** – The UTSI facility has demonstrated, in rapid succession, laboratory-scale solutions to virtually every remaining roadblock to the commercialization of MHD power production: slag separation from exhaust gases from a full slag carryover combustor; recovery of more than 90 percent of the seed, well within the amount needed for economic acceptance of the MHD/steam power plant; reduction of sulfur-oxide emissions from a coal-fired combustor to any level desired down to essentially zero; and reduction of  $\text{NO}_x$  emissions to extremely low concentrations, well below current or anticipated EPA standards. The slag separation and seed recovery experiments were carried out on two test trains: a combustor and an operational MHD generator coupled to a radiant boiler and then to a separation cyclone followed by various gas sampling devices and either a baghouse or a scrubber, and a second similar test train in which the MHD generator was simulated and had no magnet. The results indicated that seed recovery is enhanced by increased slag separation temperatures, high magnetic field application, increased system size, and washing with a dilute acid. Seed recovery is reduced by increased turbulence and long residence times. Measurements on  $\text{SO}_x$  reduction were as predicted by chemical calculations except somewhat more ( $\sim 20$  percent) than stoichiometric seed is required to reduce emissions to essentially zero. Tests on  $\text{NO}_x$  reduction were conducted using the radiant boiler and cyclone as described above, which results in a residence time of about 1.3 sec. The  $\text{NO}_x$  concentration was measured as a function of oxidant ratio in the combustor. Results were rather dramatic. As the oxidant ratio was reduced, the  $\text{NO}_x$  decreased from several thousand ppm to less than 50 ppm at a 0.85 oxidant ratio, well below the fuel-bound nitrogen level.

Design and contracting for the new test facility were accomplished. Construction is underway and completion is scheduled for January 1978. Design and procurement of components for the test trains of this facility are progressing on schedule to permit initial testing to begin in January 1979. The new facility will offer improvements in virtually every phase of test operations, but the most significant is in coal handling, providing a capability for continuous operation of any one of three bays at 8 lbm/sec total flow. A digital data acquisition system was procured and placed in operation in the existing test facility that will later serve the new facility. It provides a capability for on-line data acquisition, reduction, and display. It is capable of conversion of 200 analog data channels to digital, reduction of each channel to basic engineering units, and the display of the reduced data. System studies and material experimentation continued in an effort to further define the first-generation MHD commercial power plant. A chemical laboratory was established and staffed that has the capability in-house to do most chemical analyses required by the project.

**PLANS FOR THE COMING YEAR** – The major priority will be the completion of the construction and preparation for startup of the new test facility including continued design, procurement, and/or fabrication of components for the test trains. Work will continue in coordination with Argonne National Laboratory to define the parameters of the superconducting magnet. Work will continue on slag separation and seed collection. A new program will begin in seed regeneration—conversion of the recovered potassium compounds back to potassium carbonate and the processing or disposition of sulfur compounds. This work will concentrate on examining the various processes available in small batch experiments to choose the most promising system for on-line application in the new facility. Work will also continue in the  $\text{NO}_x$  reduction area with a vitiation heater being constructed so that air can be used as the oxidant. Also, modifications are being made in the flow train so that the residence time and temperature can be varied to find the best time-temperature profile for  $\text{NO}_x$  decomposition. Inverters will be completed so that testing of

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the generator with inverters rather than resistive load banks can be accomplished. The materials program will be expanded to include experimentation with boiler tubes made of various materials in the MHD exhaust stream to determine heat transfer and corrosion effects in this chemical environment. Testing work in electrode and insulator materials in a slag environment will continue in a search for the best generator materials.

## MHD MATERIALS AND COMPONENT DESIGNS

WESTINGHOUSE ELECTRIC CORPORATION

DOE - \$3,929,000

12/15/75 - 12/15/78

**OBJECTIVES** — This program involves the engineering design and development of long-lived high-temperature ceramic electrode systems for open-cycle, coal-fired MHD power systems. It includes the design of electrode systems, the development of improved electrode and insulator materials, and comparative testing of electrode materials and systems. A supporting objective is to extend the capabilities of testing facilities to support advances in the electrode system development effort.

**RECENT WORK AND ACCOMPLISHMENTS** — The Westinghouse Electrode System Test Facility (WESTF) is fully operational. This facility has the capability of evaluating electrode/insulator materials and systems under simulated MHD conditions for durations to greater than 100 hours. Initial operations included a series of runs with a dummy test section followed by runs with a cold-wall test section ( $\sim 500^{\circ}\text{C}$ ) and a hot-wall test section ( $\sim 1700^{\circ}\text{C}$ ). Initial operations were in the clean firing mode; subsequent operations will be in the slagging mode. Procurement of a mini-computer for use with WESTF has been initiated. Expansion of the Westinghouse MHD Test Facility (WMTF) building was completed, and removal and rearrangement of facility equipment was started in preparation for the planned facility modification. As part of the Cooperative U.S./U.S.S.R. MHD Program, the Phase III Module is being designed for testing in the Soviet U-02 facility. Extensive materials evaluation and electrode systems design effort are directed towards final systems selection for the U-02 module. This test has as its objective operation of refractory electrode assemblies (surface temperature  $\geq 1700^{\circ}\text{C}$ ) for extended durations (100+ hours) at relatively low heat fluxes ( $\approx 20\text{W}/\text{cm}^2$ ) and under clean-firing conditions. Emphasis is being placed on current leadout and electrode attachment techniques. Final selection will be based in part on the results of a series of three proof tests to be conducted in WESTF in early FY 1978. Analytical techniques include coupled thermal-structural finite-element analyses of the electrode/insulator system. An electrochemical corrosion test has been established where current is passed between two electrodes immersed in a slag-seed bath at temperatures of  $1000^{\circ}$  to  $1400^{\circ}\text{C}$ . It provides a ranking of electrode materials and can be used to demonstrate the effect of several generator conditions (current density, slag-seed composition, time, and temperature) on corrosion. Significant results are that oxides undergo high rates of corrosion as cathodes; the most promising class of materials as anodes is spinel structured oxides; the highly reducing conditions created at the cathode plus the ionic nature of the slag place very severe conditions on oxide materials although  $\text{MgCr}_2\text{O}_4$  has shown some promise as a cathode; and testing has shown that secondary phases and porosity can dramatically reduce corrosion resistance, thus, there is a need for high-purity high-density materials.

**PLANS FOR THE COMING YEAR** — Proof-testing of candidate electrode systems in WESTF will be completed followed by selection of the U-02 module electrodes for test in the U-02 facility.

Module delivery is scheduled for March, and the test for April 1978. Laboratory materials tests will continue with emphasis on materials suitable for use in coal-fired MHD generators operating in the semi-hot and hot-wall modes. Similarly, a series of materials and electrode systems tests are planned for WESTF. Installation and checkout of the mini-computer will be completed in early 1978.

### HIGH-MAGNETIC-FIELD MHD GENERATOR PROGRAM

STANFORD UNIVERSITY

DOE - \$3,383,000

7/1/76 - 9/30/79

**OBJECTIVES** — This program is investigating MHD generators operating at high magnetic fields to obtain design information. Projected MHD-steam plants for central station power plants will operate at magnetic fields higher than those that have presently been tested. As the magnetic field is increased in an MHD generator, modifications occur in the current distribution, in the boundary layer temperature and velocity profiles that affect heat transfer and friction, and in the stability of the plasma. This program involves the generation of fundamental engineering data and the dissemination of these data to the engineering community through workshops and seminars.



*Disk Generator Undergoing Thermal Test*

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**RECENT WORK AND ACCOMPLISHMENTS** – Experiments in Stanford's M-2 facility have been conducted in which fluctuations in pressure, electric field, and current were observed as a function of generator configuration and flow conditions. Various statistical techniques are used to analyze the data to obtain maximum physical insight. One of the most important results was the observation of a fourfold increase in pressure fluctuation level during operation of the generator in the Faraday configuration for the case of high-current applied magnetic field. The effect of current or magnetic field alone upon the inherent pressure fluctuation levels is slight. The frequency spectrum shows that the increase in RMS pressure fluctuation level is associated primarily with frequencies below 100 Hz. Several aspects of the experimental observations were found not in accord with earlier magnetoacoustic theories, and a new theoretical model has been developed. Predictions of the new model agree well with the observation of low-frequency pressure fluctuation levels in Faraday generators. Measurements of electron number density, temperature, and voltage drops were performed in the electrode boundary layer of the M-2 facility. Measurements were made at two different flow and electrical conditions. The boundary layer phenomena depend principally on the local current density, so no magnet was used in the experiments. Battery banks provided an applied electric field for the runs with current, and a run-in section was added to enhance boundary layer growth. Significant improvements in the signal processing techniques for the electron number density measurement have been made, allowing measurements near the wall where the signals are very weak. Improved optics and alignment procedures have increased spatial resolution near the wall, so that the first measured point in the present experiments was typically 0.4 millimeter from the electrode surface. The experimental results show that Joule heating can have substantial effects on the temperature and electron number density profiles. Near the surface, non-equilibrium is observed in the electron number density profile. Preliminary results indicate that wall roughness, probably caused by small gaps and irregularities at the electrode-insulator interfaces, may have a significant effect on the measured profiles. All of these effects can have an important influence on wall heat transfer and boundary layer voltage drops. Transverse breakdown was observed at high local current densities, giving rise to decreased electrode voltage drops and elevated electron number density near the wall. Induced and applied field experiments and theoretical calculations have been performed to aid in the understanding of Hall voltage breakdown. Comparison of theory and experiment for the applied field configuration indicates that the threshold for two-dimensional plasma and insulator thermal runaway can be taken as an upper boundary for the experimentally observed plasma and insulator dominated breakdown, respectively. To make early measurements at high magnetic fields, testing in Stanford's existing small 6-tesla superconducting magnet on disk and linear geometry has been initiated. The objective is to investigate the performance of the combustion-driven disk generator to assess its viability for commercial open-cycle power generation. The main components of the disk generator experimental facility—the combustor, the plenum chamber, and the disk generator test section—were integrated and a series of thermal tests was performed. Efforts were directed toward the development of the disk generator test section constructed with metalized MgO ceramics. The main components have undergone thermal tests of 4 hours with a mass flow rate in the range planned for the power generation experiments.

**PLANS FOR THE COMING YEAR** – Work will include experiments to test aspects of the new magnetoacoustic theoretical model. The existence and effect of transients on Hall and diagonal wall configuration generators will be investigated more fully. A numerical solution to the present analytical theory will be constructed for more exact modeling of real generators. The new theory will be extended to full-scale central station MHD generators to predict the importance of the observed unstable behavior on their operation. Full-scale operation of the disk generator facility

combined with 6-tesla superconducting magnet is scheduled for the first quarter of 1978. Investigations of the boundary layer phenomena and of the current discharge phenomena for the disk generator geometry will be continued. The performance prediction study will be directed to delineate criteria for high-enthalpy extraction at acceptable turbine efficiencies and to assess the applicability of the disk generator for a commercial MHD power plant.

## DEVELOPMENT AND EVALUATION OF MATERIALS FOR MHD POWER GENERATORS

BATTELLE, PACIFIC NORTHWEST LABORATORIES

DOE - \$210,000

4/1/75 - Continuing

**OBJECTIVES** — The goals are to develop, test, characterize, and evaluate electrodes, insulators, coal slags, and other materials related to open-cycle coal-fired MHD power generators. Materials in a MHD generator are subject to high temperatures, large temperature gradients, corrosive alkali seed, combustion products, coal slags, and electrochemical effects of high DC fields. To survive these environments requires materials with improved chemical, mechanical, physical, and thermal stabilities. These requirements vary widely within the generator system demanding a variety of nonmetallic and metallic materials; however, the electrodes and insulators of the MHD channel are of primary importance. The electrodes and insulator needs pose conflicting requirements on composition, structure, properties, and fabrication. A systematic program of materials development, testing, and evaluation will eventually result in materials which can meet these varied needs of coal-fired MHD power generators.

**RECENT WORK AND ACCOMPLISHMENTS** — Battelle (PNL) is cooperating with other laboratories in the design, testing, characterization, and evaluation of U.S. MHD channels tested in the U.S.S.R.'s U-02 facility. Phase II was successfully tested and evaluations completed. Three critical technical areas were defined: enhanced electrochemical degradation by alkali seed, attachment of ceramic electrode to metal current leadout, and correlation of materials development with channel design. Reproducible laboratory methods have been developed to duplicate and test the effects of a direct electric current and voltage and potassium seed/slag interactions on electrode and insulator materials. Scoping and screening studies on a wide variety of potential electrodes and some insulators have provided preliminary performance data to select materials with greatest potential for study. Electrochemical effects have been duplicated in the short-term laboratory tests. Comprehensive tests performed for  $\text{La}(\text{Mg})\text{CrO}_3$ ,  $3\text{MgAl}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ , and  $\text{HfO}_2$  ( $\text{Y}_2\text{O}_3$ ,  $\text{CeO}_2$ ) have provided an understanding of the enhanced electrochemical degradation in seed and slag/seed and have given direction for materials development. Electrode and insulator materials are being developed with improved electrical, thermal, and electrochemical properties including structures

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**PLANS FOR THE COMING YEAR** – Electrochemical screening and in-depth studies of oxide electrodes and insulators in slag/seed will continue, and tests of metal electrodes in coal slag/seed will be initiated. Correlation with actual MHD generation tests will be emphasized. Efforts will be initiated to develop advanced electrochemical dynamic testing methods. A study will be started to develop bonding layers between oxide electrode and metal current leadout. Development of graded electrode structures will continue. Electrodes and insulators will be characterized and evaluated in cooperation with other U.S. laboratories before and after testing of a Phase III U.S. channel in the U.S.S.R.'s U-02, including four proof tests conducted at Westinghouse. The characterization (thermal, electrical, and structural properties) of materials developed at PNL and by other U.S. contractors will continue. A study of slag layers developed in MHD electrode walls will be made.

## **SUPERCONDUCTING MHD MAGNET DEVELOPMENT**

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,  
FRANCIS BITTER NATIONAL MAGNET LABORATORY  
DOE - \$1,738,000  
5/77 - Continuing

**OBJECTIVES** – This program is developing a technological base for the design and construction of baseload superconducting MHD magnets in the late 1980's, managing the procurement of magnets for specific MHD projects, and supporting DOE in planning and directing this effort. Superconducting baseload MHD magnets must be designed with the knowledge that they will be cryostable and structurally safe. Before the design can be finalized, it is necessary to develop quantitative technology as a base. This technology development program must also involve industry in preparing to meet the needs of the baseload magnet development program. Part of the technological base acquisition program and specific model manufacturing and intermediate-scale (CDIF, ETF) magnet construction tasks will be performed by selected vendors supervised by MIT/FBNML staff in order that industry acquire essential analytical and manufacturing skills to meet the demands of baseload magnet construction on time.

**RECENT WORK AND ACCOMPLISHMENTS** – Criteria for recovery of superconductivity in composites in response to perturbing heat pulses have been defined theoretically and confirmed experimentally. Frictional perturbation is being investigated theoretically and experimentally. High-current conductors for baseload-scale magnets have been designed and will be incorporated in a winding-model magnet. A Component Test Facility that includes a 50 ft<sup>3</sup> test dewar has been constructed. Several types of major force containment structures have been evaluated analytically in preparation for an external study of superstructure. A racetrack design for the Stanford MHD magnet has been completed and a recommendation for procurement has been submitted. Conceptual designs and estimates of cost and completion time have been completed for both the conventional and superconducting CDIF magnets. The preliminary procurement effort has been completed including the dissemination of requests for proposal, evaluation of responses, and contractor selection.

**PLANS FOR THE COMING YEAR** – Procurement contracts will be issued for both CDIF magnets. A request for proposal will be issued for the Stanford superconducting MHD magnet and that magnet will be procured. Supporting analytical and verification tests will continue throughout the final design and construction phases for all magnets, with special efforts devoted to the two superconducting magnets. The CDIF conventional magnet is scheduled for installation in January



1979, the CDIF superconducting magnet (which will be the largest, highest-field superconducting MHD magnet ever constructed) in March 1980, and the Stanford superconducting MHD magnet in September 1980. In addition, a central technological data base will be established to make results of MHD and superconductivity research readily accessible.

## **REGENERATIVE HEATER DEVELOPMENT**

**FLUIDYNE ENGINEERING CORPORATION**

**DOE - \$1,046,000**

**12/20/75 - 10/15/77**

**OBJECTIVES** – The aim of this program is to develop the technology needed for high-temperature air heaters for coal-fired MHD power plants. These heaters will be of the regenerative type using ceramic material as the heat-storage medium. One specific objective is to identify ceramic materials that will resist corrosion and have high creep strength and thermal stress resistance. Another objective is to develop design criteria and/or operating procedures that will accommodate the presence of seed/slag.

**RECENT WORK AND ACCOMPLISHMENTS** – The program includes development of both directly and indirectly fired air heaters. Information from analytical studies will help guide the development program and provide size and cost estimates. Thus far, work has been on the preparation and application of analytical tools for carrying out the studies. Tradeoff studies have been completed on the effect on cost of geometric variables (e.g., cored-brick hole diameter, web thickness, bed diameter, and number of vessels) and operating variables (e.g., bed and gas temperature levels and thermal stress levels in the cored bricks). Also, preliminary information on the importance of unit cost of the heat storage material and other components on system cost has been developed. A dynamic performance analysis has been undertaken because a regenerative heater system inherently delivers an air stream whose conditions vary cyclically with time. Large variations are not tolerable, and the relationship between the heater system geometry and operating conditions (e.g., number of heaters in parallel, valve operating time, depressurization rates) and the steadiness of the delivered air stream must be determined. Study of seed/slag deposition, drainage, and removal from the heaters has been started to develop a mathematical model that can be used with subscale test data for predicting seed/slag behavior in full-scale heaters. The analysis will provide information for scaling up to large heaters. Design studies of a heater test module have been completed. A single heater will be tested under conditions that simulate full-scale MHD plant operation of a directly fired air heater and will provide a basis for the design of larger air heaters. The design support test work comprises the largest activity. Over 80 materials have been tested under various conditions of exposure to seed/slag-bearing gas streams and with gas-to-air cycling conditions simulating heater operation. These tests have shown that the magnesia-alumina and magnesia-chromia compositions have the highest corrosion resistance. Typically of 30-hour duration, the tests have been made in a subscale test rig that has recently been increased from 6 to 17 ft in length. Laboratory analyses for failure mechanisms are proceeding.

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## CRITICAL CONTRIBUTIONS IN MHD POWER GENERATION

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

DOE - \$3,786,000

6/1/75 - 5/31/78

**OBJECTIVES** – This program focuses on the investigation and solution of problems in coal-burning MHD power generation.

**RECENT WORK AND ACCOMPLISHMENTS** – The design and development of electrode/insulator modules capable of surviving and operating in the MHD environment has focused on the determination of inter-diffusion, electrical conductivity, electron emission, and phase chemistry in the  $\text{FeAl}_2\text{O}_4\text{-Fe}_2\text{O}_3$  system and on the effects of Fe contamination on  $\text{Al}_2\text{O}_3$  and  $\text{MgAl}_2\text{O}_4$ . Engineering data for reactions and eutectics between potential electrodes and insulators have been determined, and a new graded electrode,  $\text{SrZrO}_3\text{-LaFeO}_3$  has been invented. Extensive tests of electrode modules have been conducted, providing information on the soundness of different designs and the conditions under which neither arcing nor electrolytic corrosion is observed. Coal combustion studies, including both experimental work and modeling of combustor performance, have been concerned with the devolatilization yields of coals fed into a high-temperature MHD combustor, and with characterization of the behavior of the ash and of the slag layer under such combustion studies. Experimental efforts have been concentrated on determining the volatile yields from a Montana Rosebud coal pyrolyzed over a temperature range from  $1500^\circ$  to  $2500^\circ\text{K}$ . Mathematical and computational studies have addressed key phenomena affecting MHD generators, including plasma uniformities, presence of slag layer, time-dependent behavior, and interelectrode breakdown. The effects of three-dimensional inhomogeneities have been investigated; calculations of interelectrode breakdown studies were confirmed by experimental results obtained on the Soviet U-02. The theory was extended to inclined walls, which show outstanding field strength compared to flat walls. Reduction formulas for the effective, or macroscopic, Ohm's Law have been derived for inhomogeneous plasmas with anisotropic fluctuations having two general types of geometry: elongated or shortened in the direction of magnetic field, and two-dimensional, with the direction of constant properties perpendicular to the magnetic field. The derivations use the small perturbation and self-consistency methods; both methods agree well for weak nonuniformities, but differences appear at high fluctuation levels and for high values of the Hall parameter. A model for interelectrode breakdown has been used for a series of calculations aimed at constructing maps of safe and unsafe operating regions.

Experiments with a disk generator have provided some remarkable results for a disk operating with a 45-degree inlet swirl; in argon, power densities from  $100 \text{ Mw/m}^3$  at  $1950^\circ\text{K}$  to  $500 \text{ Mw/m}^3$  at  $T^\circ = 3350^\circ\text{K}$  were obtained with an enthalpy extraction of 5 percent and 17 percent, respectively. A maximum isentropic efficiency of 51 percent and a maximum electrical efficiency of 62 percent were also recorded, which are the highest power densities and efficiencies ever measured in an inert MHD generator operating in this range of temperatures. With molecular gases related to open-cycle systems, E-fields as high as 38.2 kv/m (more than a factor of 8 greater than the linear counterpart) and Hall coefficients up to 5.8 have been obtained at open circuit. Electrical efficiencies at high interaction in pure  $\text{N}_2$  exceeded 70 percent. The molecular gas tests have been conducted in both  $\text{CO}_2\text{-N}_2$  and  $\text{CO}_2\text{-N}_2\text{-H}_2$  mixtures, with the latter simulating the gaseous products of coal combustion. Nonequilibrium chemistry has been included in the data analysis, and the importance of considering chemical kinetics behavior in modeling small- to medium-scale

supersonic channels has been clearly established. One of the achievements of the component and generator modeling tasks is the design and implementation of a computer system specifically intended to permit modeling of an entire MHD plant using the techniques available to the petrochemical industry through their proprietary simulation programs.

**PLANS FOR THE COMING YEAR** – MIT's participation in the U.S./U.S.S.R. Cooperative Program in MHD power generation has included numerous aspects of the U-25 channel program, the U-02 materials program, the superconducting magnet program, and the investigation of the characteristics of interelectrode breakdown in a large-size MHD generator.

## **MHD POWER GENERATION RESEARCH, DEVELOPMENT, AND ENGINEERING**

MONTANA ENERGY AND MHD RESEARCH AND DEVELOPMENT INSTITUTE, INC.

DOE - \$7,923,000

10/1/76 - 10/1/77

**OBJECTIVES** – This project is preparing for the future operation and management of the Component Development and Integration Facility (CDIF), a 50 Mw<sub>t</sub> facility. This task additionally includes evaluating capabilities and design information required to support MHD materials selection and application and characterization of Montana coals for open-cycle MHD power generation. Also, environmental studies are being conducted to minimize the impact of MHD technology.

**RECENT WORK AND ACCOMPLISHMENTS** – Plans and procedures are being provided to DOE on facility maintenance, startup and operation, material and data control, test train and interface hardware checkout, and quality assurance and safety. Procedures also are being established for instrument and electronic equipment calibration and repair, modeling and plant simulation to predict facility operation, and software development in support of data acquisition and plant operation. Information to minimize emissions from the CDIF is being provided. This effort necessitates obtaining and validating computer models for the MHD combustion stream from the combustor through stack emissions, utilizing and reviewing sampling and analysis techniques, and characterizing growth and deposition. In the Environmental Development Plan, environmental issues and problems unique to MHD technology are being identified, and a plan will be presented that can be used to solve the problems through appropriate and coordinated research activities. Data are being obtained on the thermal transport properties of slag-impregnated ceramic refractories for use in air preheaters. Controlled structure ceramic materials are being fabricated and characterized to provide the controlled parameter changes that are required to determine reaction mechanisms in MHD components. Slag refractory wettability studies also are being conducted to model the performance of air preheaters.

Under subcontract, efforts at the Montana College of Mineral Science and Technology (MCMS&T) have provided information on coal slag compositional variations, burner design, and environmental controls. Work is focused on the type and extent of corrosion, the dimensional changes, and the alterations in room-temperature strength of the materials. At Montana State University (MSU), experimental programs are in progress to determine the broad range of physical characteristics within slag-seed mixtures, including the vapor pressures of seed and seed compounds, electrical and thermal conductivity, current carrier mobility, and thermionic emission parameters. Data on the thermodynamics and physical properties of seeded coal slag are being collected and analyzed. Other work includes specifying the data acquisition transducers necessary for monitoring

individual component behavior and integrity in a 250 Mw<sub>t</sub> plant. Data acquisition includes programs for designing and analyzing MHD steam power plants, a data flow simulation program for predicting computer data reduction requirements in an operating MHD steam power plant, and combined-cycle system simulations to determine methods of control. In addition, a computer simulation model of an MHD generator was developed.

**PLANS FOR THE COMING YEAR** — MERDI will continue to generate specific research projects that apply to MHD technology. They include defining biological effects induced by high-intensity magnetic fields associated with MHD systems, assessing the ecological impacts of CDIF emissions, and determining the health effects of trace elements and organic compounds identified in the MHD process. MERDI will prepare the more advanced plans and procedures for the safe and efficient operation of the CDIF and provide scheduling with test article suppliers. Facility startup activities will begin. Slag-seed wettability studies will be expanded to include alternate synthetic slag compositions and natural slags. Research at MCMS&T will focus on coal and its properties and preparation. Information on the physical properties of specific western coals will be analyzed, and corrosion tests on candidate materials continued in simulated combustion atmospheres. At MSU, work will begin to evaluate the effects of coal slag and seed on the performance of high-temperature regenerative heat exchangers. The analysis of seeded coal slag will continue through vapor-pressure measurements of potassium and potassium-seed compounds, thermodynamic analysis, and electrical property measurements.

## DEVELOPMENT, TESTING, AND EVALUATION OF MHD MATERIALS

NATIONAL BUREAU OF STANDARDS  
DOE - \$733,000  
10/1/76 - 9/30/77

**OBJECTIVES** — This program is providing chemical and physical data for high-temperature materials that show promise for use in coal-fired open-cycle MHD power systems. Areas in which investigations are being conducted include characterization of coal slag and its interaction with system components; development of electrode, insulator, and preheater materials that will not fail over extended periods of use; development of seed-recovery methods; determination of phase equilibria; examination of diffusion rates of seed in slag and other materials; and durability studies of prototype MHD subsystems.

**RECENT WORK AND ACCOMPLISHMENTS** — NBS is also involved in post- and pre-test characterization (with Westinghouse and Battelle NW Laboratories) of electrode and insulator modules for the Soviet U-02 MHD test channel in Moscow. Some important results from these investigations include final evaluation of test materials for U-02 Phase II; evaluations of screening tests for U-25 generator (clean fuel) conducted at Reynolds and MIT; and characterization of various electrode materials, which has indicated that MAFF-31 (3:1 MgAl<sub>2</sub>O<sub>4</sub>-Fe<sub>3</sub>O<sub>4</sub>) is a prime candidate. Results for the FluidDyne preheater testing showed that in most cases a MgO deficient layer is formed on the reacted surface of the material, for both Mg-Al spinel and RFG (rebonded chrome-magnesia refractor material). Over 100 MHD materials (powders, dense ceramics, etc.) have been structurally analyzed by X-ray diffraction techniques.

Considerable effort has been devoted to improving the fabrication of electrode and insulator assemblies to overcome the problem of the ceramic body parting from the metal cooling strip. Test

electrodes have shown that the use of arc plasma spraying for the deposition of the ceramic body gives promise of strong and durable metal to ceramic bonds. An important aspect of MHD research is the preparation of synthetic coal slags and the determination of their viscosity plus that of any real slags furnished to NBS by other contractors. A "base" slag was devised that is intermediate between Montana Rosebud and Illinois No. 6; viscosities have been determined as a function of varying amounts of  $\text{SiO}_2$ . The effect on viscosity by varying the amount of  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ,  $\text{CaO}$  and  $\text{MgO}$  is being determined. Vaporization studies have provided vapor pressure data on  $\text{K}_2\text{SO}_4$  and distribution of K-species in the plasma and slag, and information on the various K-compounds that may form in the slag layer on the channel wall. Interaction of slag, seed, and MHD generator components has been studied by means of the determination of the phase relationships between potassium oxide and slag components ( $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{FeO}_x$ - $\text{CaO}$ - $\text{MgO}$ ) as well as the reactions involving K-compounds with various electrode materials ( $\text{ZrO}_2$ - $\text{CeO}_2$ ,  $\text{LaCrO}_3$ , and different spinels). Results of diffusion couples (electrode-insulator-electrode) experiments have given concentration profiles of Fe from MAFF-31 (electrode) into various insulator materials thereby suggesting the relative insulating properties of different materials for various temperatures and times. Electrical conductivity measurements have been made on many candidate electrode materials. Of these, iron-doped spinels are promising candidate electrodes particularly for a slagging system. They possess both high melting points and good electrical conductivity at MHD operating temperatures. Data on the mechanical properties, nominal chemical composition, and hot corrosion resistance of many metal alloys that appear promising in MHD-type service have been collected and tabulated. These alloys include stainless steels, inconels, and Hastelloys among others.

**PLANS FOR THE COMING YEAR** — There will be a continued major effort in the area of post- and pre-test characterization of the candidate MHD materials tested under various MHD test conditions. Work will continue to study the seed-slag-component interaction via phase analysis. These efforts will be combined with work on the characterization, fabrication, design, and testing of electrode insulator materials.

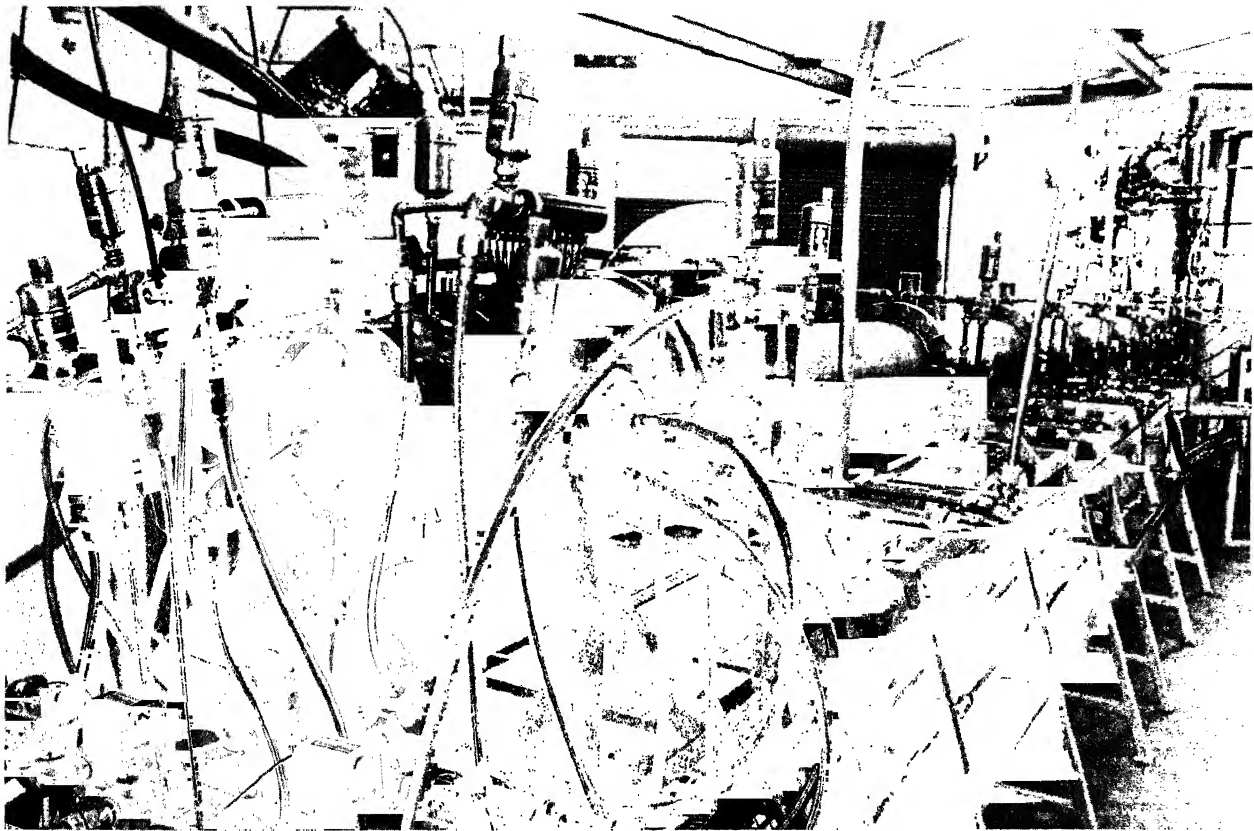
## TESTING AND EVALUATION OF MHD MATERIALS AND SUBSTRUCTURES

ENERGY RESEARCH CENTER, MISSISSIPPI STATE UNIVERSITY

DOE - \$1,085,000

4/8/76 - 4/7/79

**OBJECTIVES** — This work is generating design data pertinent to MHD radiant boilers, air heaters, and steam superheater components. The test train will simulate gas-seed-slag conditions in the coal-fired baseline MHD plant, characterize the component environments, and determine potential failure mechanisms and their solution for the radiant boiler and steam superheater regions. The slag-wall characteristics will also be studied for chemical composition down the gas path and for



*Test Stand for Simulation of Conditions in a Coal-Fired Baseline MHD Plant*

**RECENT WORK AND ACCOMPLISHMENTS** — Test train design, including automatic control and monitoring, has been completed, and construction is nearing completion. Shakedown and calibration programs have been written and checked out. The computer code NASA CEC-76 was used to calculate chemical equilibria for mixtures consisting of seed, slag, fuel oil, and air for temperatures from 4850° to 2000°F, which are required in thermal analysis of the flow train. The thermal analysis code then predicts such parameters as longitudinal gas-temperature profile, surface temperature of slag deposit on pipe wall, velocities in flowing slag layer, pipe-wall temperatures, and heat-transfer rates. This code is being used in system design and in interpretation of experimental results to provide engineering data required for the design of larger systems. Diagnostic instrumentation is employed to measure a number of critical parameters in the thermal model, and sensitivity analyses have been developed to indicate anticipated errors involved in the diagnostics.

**PLANS FOR THE COMING YEAR** — Before initiating tubular radiant boiler tests, tests will be made with water-cooled annular boiler sections (concentric cylinders) to determine the transition cooling section length needed to cool the gas down to 3800°F, and to design the tubular radiant boiler test section. A radiant boiler will be designed next for inclusion in the test section where the temperature of the gas stream drops from 3800° to 3000°F.

## TECHNICAL SUPPORT FOR THE OPEN-CYCLE MHD PROGRAM

ARGONNE NATIONAL LABORATORY

DOE - \$780,000

11/74 - Continuing

**OBJECTIVES** – This work is providing technical evaluations of programmatic activities for DOE's MHD Division staff and developing and applying system design capabilities for guidance in long-range planning. These system studies serve to identify potential technological problems at an early stage in concept development; determine the probable performance of various options and configurations for open-cycle MHD power plants; quantify economic and environmental impact; provide pertinent technical information in developing MHD; and aid program planning for various experimental facilities. Systems analysis computer codes are to be prepared and used as requested to assist the MHD Division in optimizing the open-cycle MHD plant design with respect to performance and cost. Specialists from other in-house MHD programs fulfill specific requests made by the MHD Division for assistance in its program monitoring and evaluation functions. The program planning for new facilities is of the utmost importance to provide for the timely acquisition of test results needed to develop open-cycle plants.

**RECENT WORK AND ACCOMPLISHMENTS** – The systems analysis computer code developed for studying MHD steam-power-generating systems is fully operational. System studies were carried out for several different proposed cycles including investigations of alternate reheat arrangements of the proposed Engineering Test Facility (ETF). Development of the individual subroutines has continued, with the inclusion of the more detailed analysis of the combustion process and the seed-slag interactions in downstream components. Optimization studies using the recently completed executive code were initiated and are leading to an improved systems code through the definition of the important coupling effects between components within the system. The nozzle, MHD generator, and diffuser computer codes were extended to account for two-dimensional effects. While the code will continue to be upgraded, it is presently one of the most complete analytical programs available. A disk generator code has been developed and more sophisticated optimization methods have been incorporated. A method was also developed to account for arc discharges at the electrodes.

Monitoring of contractor performance and review of equipment design and construction have been continued. Reviews of contractor progress reports and work plans for most of the contracts have been supplemented by site visits. Design reviews have included evaluation of three coal-fired combustor concepts that were candidate systems for the CDIF and three MHD generator designs also intended for the CDIF. ETF design studies were initiated with three commercial contractors in February of 1977. The ANL contribution to these studies has been to assist in review of technical documents developed (i.e., ETF Design Criteria Document); participation in interim design reviews at the contractor's offices; and assessment of the accuracy of the analytical models used by the contractors in their MHD channel designs. In the last case, calculations were performed using computer codes to determine where discrepancies existed. ANL, along with NASA-Lewis Research Center, has also provided direct assistance to the DOE/MHD staff in planning and organizing the later phases of the ETF design development program.

**PLANS FOR THE COMING YEAR** – Refinement of the computer codes for MHD plant performance and optimization will continue. Emphasis will be placed upon methods to perform



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cost analysis of all subsystems and components. In addition, there will be a continuing effort to upgrade the analysis for components such as superheaters, reheaters, air preheaters, economizers, radiant boilers, coal handling and processing, and the second-stage combustor. The MHD generator and diffuser code development will continue, to account for additional two- and three-dimensional effects and to allow for computation with flowing slag-layers and time-dependent factors. Additional combustion kinetics codes will be developed and refined.

## MHD BALANCE-OF-PLANT PROGRAM

ARGONNE NATIONAL LABORATORY

DOE - \$300,000

6/75 - Continuing

**OBJECTIVES** – The balance-of-plant (BOP) program is providing the engineering basis for designing and operating the downstream gas system; the combustor feed systems; and the seed recycling process. The gas system downstream of the MHD channel and diffuser is analogous to the steam generating plant in a conventional power plant, but it must perform the additional functions of separating slag and seed, recovering nearly all of the seed material, preheating air to high temperatures, and controlling  $\text{NO}_x$  and  $\text{SO}_2$  concentrations at acceptable levels. In addition, the MHD downstream gas system must operate under thermal and corrosive conditions that are more severe than those in conventional plants. Another objective is to support the design of the MHD Engineering Test Facility (ETF) downstream components. ANL will prepare a national BOP program plan and serve as a technical program coordinator. The program makes maximum use of existing boiler technology and allied industrial experience. It will supplement that experience by obtaining additional data and information needed for the practical design of large-scale downstream components. The use of various U.S. facilities to aid this effort is to be recommended. Additional tests may be made at ANL to provide information not attainable elsewhere; check or corroborate critical information obtained by DOE contractors; and verify proposals or assumptions made by contractors.

**RECENT WORK AND ACCOMPLISHMENTS** – A national program plan for attacking the technological BOP problems was drafted in cooperation with the DOE/MHD staff, and primary problem areas were identified. The general strategy for solving these problems is the design, construction, and operation of prototypes of key system components—radiant boiler, superheater, steam reheater, and seed regenerative components. Radiant boiler concepts similar to conventional radiant furnaces with slagging walls have been analyzed and found to meet the requirements for an MHD plant. The water-cooled walls are covered by a steady-state slag film, which protects the steam tubes and limits the heat flux to conventional levels. The loss of seed to the slag is low because the slag surface temperature is high. This component may operate at conditions to achieve a reduction of  $\text{NO}_x$  concentration acceptable to EPA. The analytical model developed for the radiant boiler provides a sound basis for design of experiments and of the full-scale equipment. Analytical modeling of the contribution of suspended particles to the thermal radiation field was started. The absorption and scattering of radiation by the particles and the gas provide heat transfer estimates for large-scale units. A variation of the particle formation model can be applied to the sections of downstream gas system where seed condenses. Means were developed for estimating seed removal by vapor condensation and particle collection by turbulent and thermophoretic forces. Seed condensers are not being developed elsewhere in the United States. A computer model that estimates the seed-slag condensed phase compositions in equilibrium with typical MHD combustion



gas was completed. Calculated and experimental results agree within the accuracies of the property data.

**PLANS FOR THE COMING YEAR** – A task force will be formed to make maximum benefit of practical industrial experience in the design of downstream boilers, superheaters, reheaters, preheaters, and seed recovery equipment. This group will identify facilities and critical areas for which experimental or scaling data are needed, and prepare test plans for the needed work. Contracts for conceptual design studies of the radiant boiler and superheater-seed condenser will be awarded to commercial vendors. Experimental facilities will be completed to obtain the data to support the development of analytical models. A program to select ceramic and metallic materials for service in the downstream gas system will be continued. The immediate goal is the selection of materials for the prototype components. Tests will be made in the 60-kw plasma torch facility and in other available apparatus. Newly completed apparatus will be used for measuring gas-slag-seed equilibrium compositions. These data will be used to improve the thermodynamic models that can be used to predict with greater confidence the behavior of seed and construction materials in the MHD system. Several processes for regenerating seed material will be evaluated, with the objective of selecting promising techniques for further development. For many of the processes, the need is for information on reaction rates. Small-scale experiments to obtain the kinetic data needed for the design of larger scale process equipment will be started.

#### **ARCHITECT/ENGINEERING SUPPORT FOR MHD TEST FACILITIES**

GILBERT ASSOCIATES, INC.  
DOE - \$2,447,000  
3/15/76 - Continuing

**OBJECTIVES** – Specified architect/engineering, scientific, and economic consulting services in support of the DOE Fossil Energy Magnetohydrodynamics (MHD) Division are provided. These efforts should develop high-efficiency power-generating plants that utilize clean applications of our most abundant fossil energy resource—coal. Specific services deal with the review and evaluation of MHD power test facilities and systems, subsystems, and system components. Planning, scheduling, and management information support for test facilities are also provided.

**RECENT WORK AND ACCOMPLISHMENTS** – For the 50 Mw<sub>e</sub> Component Development and Integration Facility (CDIF) in Butte, Montana, detailed design reviews of all Title II engineering were carried out including process flow sheets, equipment specifications, construction specifications, and construction drawings. Scale models were constructed representing the CDIF site and the Test and Operations Buildings. In addition, studies were completed on the feasibility of increasing the number of test trains in the CDIF facility. Detailed design reviews were also performed for the 250 Mw<sub>t</sub> MHD Engineering Test Facility, the Coal-Fired Flow Facility located at the University of Tennessee Space Institute, and the Stanford Magnet Facility. Partial revisions and updating of the June 1976 baseline plant System Design Description (SDD) were carried out, and "A Reference Summary of Current MHD Test Facilities and Their Capabilities" was updated. MHD advanced design subsystems and components for which technical or economic analyses were undertaken included the combustor, seed regeneration/replacement, high-temperature air heater, reheat channels, oxygen enrichment, and separately fired air heaters. A digest was prepared of utility practices and policies in the maintenance of large, coal-fired, baseload steam power plants to

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be used in developing similar requirements for future MHD power plant designs. Administrative tasks were carried out in support of the International Cooperative Program, a procurement planning system, and computerized financial data base program. Unsolicited technical and scientific proposals to the MHD Division were evaluated.

**PLANS FOR THE COMING YEAR** — Work in Architect/Engineering support will continue.

### **MHD-ETF CONCEPTUAL DESIGN**

**WESTINGHOUSE ELECTRIC CORPORATION**

**DOE - \$785,000**

**1/31/77 - 1/31/78**

**OBJECTIVES** — The MHD-ETF conceptual design study is defining a pilot-scale coal-burning open-cycle MHD plant. Supporting this primary objective are the following secondary ones: determine a power rating for the plant that will allow predictable extrapolation upwards to a commercial-size plant, and logical extrapolation and progression in size and technology from components and processes that can be tested in existing and planned MHD component test facilities; determining the criteria for a pilot-scale demonstration plant, and the most appropriate selection of components and plant design to meet these criteria; and providing sufficiently detailed information on a selected or reference design in the areas of component and process design, plant design, program requirements, scheduling and cost to serve as a basis for budgeting and planning, and to allow proceeding to preliminary design. The eventual development and testing of the ETF will provide demonstration of a higher efficiency method of converting coal energy to electrical energy than presently available in central power plant practice and will provide information necessary to the design of commercial plants.

**RECENT WORK AND ACCOMPLISHMENTS** — To guide the conceptual design study effort, a criteria document has been prepared for the ETF facility that follows usual practices in planning, design, and construction of major pilot-scale development power plants. It serves as a basis for a document, upon completion of the conceptual design, that will permit understanding of program and guidelines as the project progresses. A conceptual design has been conducted of three potential configurations of ETF: a scaled plant following the general configuration of a baseline commercial plant previously prepared under DOE contract that was scaled to the minimum size reheat turbine-generator set commercially available; a plant without a turbine-generator but of a size useful in developing and testing the MHD components and system in a minimum size that can be extrapolated to commercial units; a plant initially constructed to test major MHD components and expandable in a progressive manner to incorporate additional components and systems until finally a totally integrated power plant configuration exists (this plant was sized to be compatible with the minimum size non-reheat turbine-generator set commercially available). In addition, technical evaluation, cost and schedule information has been prepared for each concept.

**PLANS FOR THE COMING YEAR** — Based on the study of the three conceptual designs, a single reference design will be prepared and recommended for DOE consideration. This reference design report will contain recommendations of power rating, configuration, technical features, supporting development programs, schedules, and cost.

## MHD TEST FACILITY, PLANT DEFINITION, AND TECHNOLOGY ASSESSMENTS

NASA LEWIS RESEARCH CENTER

DOE - \$397,000

7/18/77 - Continuing

**OBJECTIVES** – This work is providing the engineering analyses and technology assessments for rapidly and cost-effectively commercializing MHD/steam-cycle electric power plants. This effort requires the definition of conceptual designs for early and advanced plants and an assessment of the technology required to build them. A complementary objective is to define test facilities and/or pilot plants that optimally develop the missing technologies and satisfy intermediate milestones. The demonstration of the commercial readiness of MHD involves the synchronized development of several high-technology components and the scale-up or validation of many others. The substantial costs and lead times of these programs demand accurate planning. This effort will provide the requisite understanding of the numerous possible tradeoffs between components and the effects of various plant configurations.

**RECENT WORK AND ACCOMPLISHMENTS** – An interdisciplinary team has been formed by drawing on Lewis's discipline-oriented divisions including power systems analysts; facility engineers; computer specialists; and experts in MHD, combustion, heat exchangers, and materials. The team is reviewing and contrasting the results of three parallel ETF conceptual design studies to identify the important technical issues and estimate the developmental risks. It is also assisting the MHD Division of DOE by participating on various advisory and review panels. The team members have completed an evaluation of the status of technology in their disciplines as it pertains to MHD and have reviewed the existing development programs.

**PLANS FOR THE COMING YEAR** – A contract is being issued to a team composed of a power plant supplier, an A&E, an MHD expert, and others for cost and performance studies of specific commercial-scale MHD power plants. In-house performance studies will be used to help define and guide the contracted work and to generalize the results. Existing computer analysis techniques will be modified or adapted as required. The results of the technology assessment are now being used to identify which components require further modeling and development of a data base for evaluation in system studies. Contracts to generate this information will be issued as appropriate.

## COMPUTER ANALYSIS AND EVALUATION OF MHD SYSTEMS

STD RESEARCH CORPORATION

DOE - \$4,498,000

3/1/76 - 2/28/79

**OBJECTIVES** – This project involves the performance of analyses and evaluation of proposed and operating MHD generators and the provision of a detailed systems analysis of complete, baseload MHD power systems utilizing coal, by use of available computer codes. The work is directed toward the most efficient scaleup of MHD generators, components, and systems through the use of realistic analytical computer models of MHD phenomena, channels, and complete central power stations.

**RECENT WORK AND ACCOMPLISHMENTS** – Various operating MHD channels, including the MK-VI (C) (D) and U-25-C in normal and open-circuit operation have been analyzed. Good agreement with experiment has been obtained by use of the previously available codes. Very large

open-circuit channel voltages and boundary-layer separation in the U-25—C were shown to be a potential operational problem. Transient flow analyses of the AEDC-HPDE MHD flow train have been carried out for the proposed starting and shutdown control sequences. Results indicate the need to schedule the opening of valves in a fail-safe manner to maintain the structural integrity of the experimental facility. Several detailed studies of U-25 system arcing stability, power takeoff cable/diode loading/thermal dynamics, nonuniform entrance conditions, and test data acquisition requirements to obtain analytically correctable data have been carried out. Study of three-dimensional effects on channel performance is being pursued, as are computations of the electric/current density fields in the end region of an MHD channel to investigate the effect of gas-dynamic coupling. The effect that aluminum oxides may have upon the conductivity of an MHD plasma has been a point of contention. Analysis showed that the effect of  $\text{AlOH}_2$  on electron concentrations in the channel under MHD conditions is negligible regardless of the value chosen for its heat of formation. There has also been speculation as to the existence of  $\text{AlO}_2$  under conditions in the MHD channel. Previous calculations had shown that, if present,  $\text{AlO}_2$  could serve as a significant sink for electrons. The applicable original papers were reviewed in detail. There appears to be good evidence that  $\text{AlO}_2(\text{g})$  would exist under channel conditions. Inputs to the STD Research code have been updated with variable inflation data to improve the quality of the component cost and performance data base, the simulation of MHD systems, and the usefulness of outputs. Agreement between code results and actual operating data is excellent. Emissions from coal-fired MHD power plants are being characterized. An analysis was completed on the effects of projected mid-1980's air emissions standards for  $\text{SO}_2$  and  $\text{NO}_x$ . The projected threefold reduction in  $\text{SO}_2$  (to  $0.4 \text{ lb SO}_2/10^6 \text{ Btu}$ ) would require additional energy for removal of sulfur during seed regeneration. The effect would be a reduction of about 0.5 percent in efficiency of the DOE baseline  $1000 \text{ Mw}_e$  MHD plant. Projected  $\text{NO}_2$  standards of  $0.14 \text{ lb NO}_2/10^6 \text{ Btu}$  (compared to the present  $0.70 \text{ lb NO}_2/10^6 \text{ Btu}$ ) will require additional control efforts. Projected DOE baseline plant emissions are  $0.66 \text{ lb NO}_2/10^6 \text{ Btu}$ . Combustion modification or increased residence times in the radiant boiler and high-temperature air heater (to promote thermal decomposition) appear not to be sufficient to reduce  $\text{NO}_x$  levels to the projected standards. Some form of stack gas treatment seems to be necessary to meet mid-1980's  $\text{NO}_2$  emission standards.

**PLANS FOR THE COMING YEAR** — Work will continue on assessments of channel performance and operation, on application of the available analyses of detailed fluid mechanics, heat transfer, and electromagnetic phenomena to existing MHD channels. The code structure will undergo further testing, and a final structure selection to meet all goals of the program will be implemented by mid-1978. The component modeling inputs and the input data base will be updated to reflect available information on component types, design criterion, and cost characteristics. Work will continue defining environmental effects of MHD plants. Analysis of  $\text{NO}_x$  control methods, trace element emissions, and seed loss in slag will be completed.

## CHARACTERIZATION OF OPEN-CYCLE COAL-FIRED MHD GENERATORS

AERODYNE RESEARCH, INC.

DOE - \$289,000

7/1/76 - 12/31/77

**OBJECTIVES** — Coal is a complex chemical substance containing variable amounts of metallic and nonmetallic substances in addition to the desired carbon and hydrogen fuel elements. An understanding is being sought on how these nonfuel components of coal will affect the electron and

alkali seed chemistry in high-temperature coal combustion systems like those envisioned for direct-fired MHD generators.

**RECENT WORK AND ACCOMPLISHMENTS** – Three specific problems have been considered. The first is to characterize the formation of negative ions resulting from electron attachment processes in the combustion flow. The second area involves the role slag condensation may play in determining the electron density through recombination, also adversely affecting conductivity in the core flow. The competitive balance between thermionic emission from slag droplets and electron/ion recombination on the droplet surfaces may be severely tipped in favor of electron loss processes, depending on the slag properties. The third area is the heterogeneous interaction of alkali seed with particles formed by slag condensation in the generator channel. Alkali seed material can be chemically bound into the molten slag particles tightly enough that seed recovery becomes prohibitively expensive. The loss of significant amounts of alkali seed to the slag could have a serious economic impact on proposed MHD systems. A coupled approach, involving both theoretical modeling and experimental measurements, has been devised to explore the negative ion formation, the electron/slag interaction, and the alkali/slag interaction problems. The overall goal is to devise and validate reliable theoretical models. To date, a one-dimensional model of the fluid and MHD processes in the channel core flow has been completed. This model includes subprograms for the calculations of the equilibrium chemical composition and the plasma conductivity at arbitrary stations in the core flow. An extensive literature search to gather information on the structure and thermochemistry of suspected negative ion species has been completed, and thermochemical models of approximately 30 negative ion species have been added to the equilibrium composition code. Extensive equilibrium code runs have been performed to identify the impact of coal type and design parameters on generator efficiency. Calculations of plasma properties with this code are now being analyzed to identify possible bottlenecks in either the negative ion or slag condensation kinetics. Formulation of a finite rate description of key chemical kinetic processes has been completed and added to the model. Additional modeling efforts have identified and quantified a potentially serious conductivity loss mechanism for full-scale open-cycle MHD generators. This mechanism involves fast electron/ion recombination induced by plasma interactions with vaporized slag condensation nuclei.

**PLANS FOR THE COMING YEAR** – The acquisition of laboratory data and minor computer code modifications will be accomplished. The computer code will then be documented and made available to the MHD research community. A preliminary design of a mass spectrometric probe for use in full-scale MHD facilities will be completed.

## **MECHANICAL PERTURBATION STUDIES FOR LARGE MHD SUPERCONDUCTING MAGNETS**

ARGONNE NATIONAL LABORATORY

DOE - \$97,000

4/77 - 9/78

**OBJECTIVES** – This project is seeking to understand the stability of large MHD magnets, develop an analytical model to predict the recovery current of magnets, and validate the model and provide needed data on stability. Mechanical perturbations have been selected for study, based on working experience as to the major source of heat pulses into large, superconducting magnets.

**RECENT WORK AND ACCOMPLISHMENTS** – A program to develop basic information on the effect of local mechanical perturbations on cryostatic stability is underway. An analytical model for

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computing the transient recovery following the mechanical perturbation has been developed. A test program has been undertaken to develop data needed to verify the conclusions reached through the study. Mechanical perturbation measurements on the U.S. SCMS superconducting MHD magnet have also been analyzed. This magnet has successfully achieved full design field of 5 T at a current of 892A, corresponding to a joule heating per cooled area of  $7 \text{ kw/m}^2$ . The principal concern for the coil stability was the frictional heating associated with conductor motion. Two methods were used to detect the mechanical motion: one involved the use of accelerometers mounted on the magnet vessel; the other the use of potentiometers connected across the magnet terminals. It was found that the second method was very sensitive, and the information obtained proved exceedingly valuable in choosing the charging rate.

**PLANS FOR THE COMING YEAR** – The analytical model will be validated against experimental results. Recent measurements on transient heat transfer will be incorporated if appropriate. The experimental program will be completed. Those experiments that suggest a parameter dependence will be repeated with different values of the parameter; for example, the vapor locking experiments will be repeated with different channel sizes and orientations. The recovery current experiment will be repeated with provision for inter-turn heat conduction.

## NUMERICAL CODES FOR MHD FLOWS

SANDIA LABORATORIES  
DOE - \$150,000  
7/1/76 - Continuing

**OBJECTIVES** – Numerical techniques and codes for characterizing coal-combustion-operated MHD generators and diffusers are being developed to provide the MHD Division with evaluative guidelines in selecting and exercising channel codes and to provide users with codes to design MHD channels/diffusers and evaluate their performance. Flow fields in MHD channels are ionized, three-dimensional, often particle-laden with multiple species, and subject to electric and magnetic fields. A complete simulation of such complex flows exceeds the capabilities of existing computers. Proper modeling, however, can incorporate most of the essential features of MHD generators and provide realistic simulation, which in turn will lead to more efficient utilization of fossil fuels such as coal.

**RECENT WORK AND ACCOMPLISHMENTS** – The development of the entry boundary conditions for a real gas has been completed for the implicit finite-difference code. For supersonic flow, a boundary condition at the nozzle throat has been determined for the real gas case. An investigation of transient startup of a channel flow has been made. When the total pressure is increased smoothly, both the explicit and implicit methods give reasonable and stable results. Improvements have been made in the iteration procedure for the initial profiles and in the distribution of the grid points across the channel. Accuracy studies have been made and show that the difference scheme is second-order when a sufficient number of grid points are used across the channel. The two-dimensional electrodynamics code calculates the current and potential fields in an expanding section of an MHD generator channel. An option to use either periodic or insulating boundary conditions at the inflow and outflow boundaries was added. The code can now be used for both Faraday-connected and diagonal-connected channels. The slag vapor condensation problem for laminar boundary layer flow over infinitely segmented electrodes in an MHD channel has been extended to include a finite-thickness liquid-slag layer. Numerical solution of the slag layer equations is nearly complete.

**PLANS FOR THE COMING YEAR** – The unsteady one-dimensional channel code will be test run extensively. The coupled two-dimensional real gas flow and electrodynamics code will be tested against experimental data obtained in various MHD channels. An iterative procedure will be developed to couple the slag layer solution with the gas-phase boundary layer solution at the interface. Arc formation and migration studies near electrodes will continue by exercising the time-dependent version of the coupled two-dimensional real gas flow and electrodynamics code. The results will be checked using two other independent approaches.

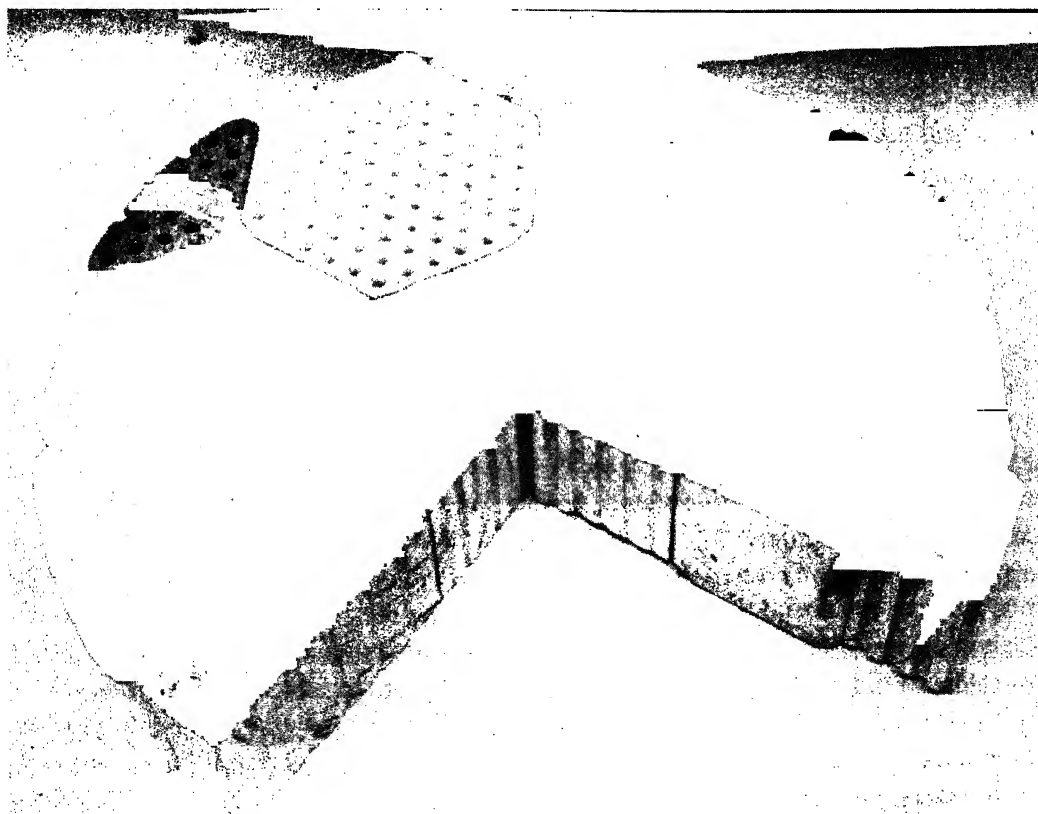
## **CLOSED-CYCLE NONEQUILIBRIUM MHD POWER GENERATION WITH DIRECT COAL FIRING**

**GENERAL ELECTRIC SPACE SCIENCES LABORATORY**

**DOE - \$2,199,000**

**4/10/76 - 3/31/79**

**OBJECTIVES** – This program is examining and evaluating closed-cycle nonequilibrium plasma MHD (CCMHD) as the topping portion of a direct coal-fired MHD/steam power cycle. This work involves continuing experimental characterization of the combustion gas and particulate contamination of the noble gas working fluid resulting from the high-temperature regenerative heat-exchange process; conversion of the existing ceramic-regenerative heat-exchanger test facility to direct coal firing with appropriate hot valving allowing simulation of the front end of the CCMHD power plant; experimental support analysis including fluid-mechanical cyclone-combustor design and slag-flow and deposition modeling; analytical determination of molecular contaminant effects on CCMHD generator performance; and CCMHD systems analysis and optimization.



*Top of Heat Exchanger Matrix Following Slag Injection*



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**RECENT WORK AND ACCOMPLISHMENTS** – The ceramic regenerative heat-exchanger molecular gas contaminant carryover testing was completed with encouraging results. It was found that the refractory insulation and heat exchange matrix used for these tests did not substantially affect the storage of molecular gases and would allow average contamination levels to be delivered to the MHD generator that were below levels believed to cause significant degradation of plasma conductivity because of suppression of nonequilibrium ionization. Gaseous fuel, reheat duration, and evacuation pressure were parametrically varied during this program with the result that only evacuation pressure had a significant effect on contaminant level and that contaminant levels were nearly linearly related to evacuation pressure. Preliminary investigations of the effects of flyash carryover into the heat-exchanger matrix were made by injecting Montana Rosebud flyash into the propane burner's combustion air at a rate simulating 10 and 25 percent ash carryover. For the limited period of operation in this mode and with intermediate argon blowdowns, the ash either soaked into the somewhat porous refractory at the top of the matrix as liquid slag or passed completely through with no tendency for radial deposition in the flues. Conversion of the heat-exchanger test facility to direct coal firing is in the final construction phase. The heat-exchanger matrix is being modified to a more slag-resistant alumina with larger gas passages to accommodate slag carryover.

Experimental support analysis has resulted in a novel air-cooled cyclone combustor design including predictions of slag-retention capability. Channel performance modeling has continued with development of a detailed model for molecular-gas contaminant behavior in a nonequilibrium noble gas plasma resulting in steady-state calculations of the degradation of channel performance as a function of concentration for  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{N}_2$  and  $\text{H}_2\text{O}$ . It was concluded that observed levels of contamination can be tolerated. Technical correspondence has been established with the Dutch CCMHD program resulting in review of their shock-tunnel channel performance data achieved with specific known molecular contamination levels. These data have been encouraging and have shown the primary effect of contaminants to be kinetic in terms of relaxation distance requirements. Further systems analysis has resulted in a more highly optimized cycle configuration with the bottoming plant output totally dedicated to argon compressor drive and with pressurized fluidized-bed coal gasifiers providing clean fuel gas to dome-fired refractory regenerative heat exchangers as the cycle's heat input subsystem, and in overall station efficiency of 46 percent, coal-to-busbar. Costing and plant layout have been performed on this configuration.

**PLANS FOR THE COMING YEAR** – An extended duration (~80 hr) flyash-injection heat-exchanger test is planned using the original 1/4-in. flue-size refractory matrix, involving steady-state reheat and air blowdown operation, limited by satisfaction of equilibrium slag deposition in the refractory matrix. It will use an existing refractory matrix and provide "worst case" slag-fouling information prior to final specification of a new flue size. Then, the horizontal air-cooled cyclone combustor will be brought to operational status along with its diagnostic systems. CCMHD generator analysis will continue with emphasis on the introduction of a kinetic model for contaminant behavior in the current one-dimensional generator analysis affording direct comparison with the Dutch shock tunnel results. Effort will be expended toward assembling a workable two-dimensional model that will allow incorporation of electrode, boundary-layer, and flow-mixing effects. Systems analysis will be updated, emphasizing maximum overall system simplicity with minimum cost of electricity while maintaining the level of overall cycle efficiency.



## CLOSED-CYCLE MHD FOR POWER GENERATION

THE RAND CORPORATION

DOE - \$325,000

3/31/76 - 1/15/78

**OBJECTIVES** — An independent evaluation of closed-cycle MHD power-generation systems is being conducted in the context of the state of the art of MHD technology, including open-cycle MHD, to provide the MHD Division with a sound basis for the formulation of programs and policies.

**RECENT WORK AND ACCOMPLISHMENTS** — The assessment of the closed-cycle noble gas MHD system has been completed. Two briefings were presented to MHD Division/FE during the months of September and October 1976. Work is in progress to evaluate the liquid-metal closed-cycle MHD power-generation system. A study team consisting of internationally known authorities was organized and leading members of the nation's MHD community were invited to present and discuss various issues and findings in liquid-metal MHD.

**PLANS FOR THE COMING YEAR** — Findings produced by the Rand study team on liquid-metal MHD will be coordinated, collected, and organized. Briefings will be presented, if appropriate, and study results documented.

## LIQUID-METAL MHD ENERGY CONVERSION

ARGONNE NATIONAL LABORATORY

DOE - \$300,000

3/76 - Continuing

**OBJECTIVES** — Liquid-metal MHD (LMMHD) is one of the three types of MHD energy-conversion systems being investigated. The LMMHD program will determine the performance of two-phase LMMHD generators under realistic operating conditions, develop methods to predict and optimize generator performance, and determine the performance of energy-conversion systems using this type of generator. This project is designed to complete fabrication of the first experimental generator and to perform the initial tests on it.

**RECENT WORK AND ACCOMPLISHMENTS** — The high-temperature sodium-nitrogen facility for testing LMMHD generators has been fabricated and successfully operated. The facility provides controlled streams of sodium and nitrogen to the generator, and records the parameters necessary to characterize generator performance. The facility system design description has been completed. Tests were made with a dummy test section (a straight piece of 4-in.-diameter pipe) to check out the facility and its operation, and to obtain preliminary data on the behavior of a two-phase mixture in a magnetic field as a function of temperature. Single- (sodium) and two-phase (sodium-nitrogen) pressure gradients and voltages were measured for various operating temperatures and system parameters. Analysis of the single-phase MHD pressure-gradient data revealed it to be closely correlated with existing laminar MHD theory. A simplified two-phase MHD correlation was developed that best describes the two-phase pressure-gradient data. Single-phase voltage profiles were found to be symmetric with respect to the electromagnet pole-face centerline, while two-phase profiles exhibited asymmetries resulting from the expansion of the mixture's gaseous component along the test section length. Construction of a high-temperature LMMHD generator has been

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completed, the channel successfully tested hydrostatically, and the mixer-generator test section assembled. The test plan for determining the generator characteristics has been revised.

**PLANS FOR THE COMING YEAR** – Instrumentation of the generator channel will be completed, and then generator tests performed over a wide range of flow parameters. Initially, open-circuit tests will be conducted, followed by tests at one or more load resistances. Particular emphasis will be on the effect of temperature on generator performance. Studies to determine the performance of LMMHD energy-conversion systems will continue.

## MHD CHANNEL FOR TESTING AT SOVIET U-25 FACILITY

ARGONNE NATIONAL LABORATORY

DOE - \$3,826,000

6/75 - Continuing

**OBJECTIVES** – This project includes the design, fabrication, and assembly of an open-cycle MHD generator to be tested in the U-25 facility, located at the Institute for High Temperatures in Moscow. The performance characteristics of a large-scale two-terminally-loaded, finely segmented, windowframe MHD channel will be determined to provide MHD generator operating experience on a pilot-plant scale. A successful demonstration of a long-duration generator channel will be a step toward establishing engineering feasibility of commercial MHD electrical power plants.

**RECENT WORK AND ACCOMPLISHMENTS** – Detailed designs have been completed for the components that interface with the Soviet system; namely, the nozzle, diffuser, steam-generator adapter, and water-cooling system. The design of the channel is close to completion, but it cannot be finalized until the electrode system has been specified. A computer-graphic technique for producing the windowframe drawings to scale has been developed that will provide the channel manufacturer with a separate drawing for each frame and also allow a quick check of consistency of abutting frames. The effort to develop ceramic electrodes for the channel involved several contractors knowledgeable in the area of ceramic electrode systems for MHD application. Each of the contractors provided his best electrode systems in accordance with ANL specifications, most of which were tested under a separate contract with Reynolds Metals in their facility. The tests indicated that none of those electrode systems was acceptable for the U-25 channel. Headquarters staff then assumed responsibility for defining an electrode system for the U-25 channel and launched a "crash" program involving several experts working in concert to provide applicable ceramic electrodes. The resulting candidate designs were tested, again with inconclusive results, particularly for application in the present windowframe design.

In contrast, the situation in regard to cold-wall, metallic electrodes appears much more promising. Effort now being undertaken to establish the U-25 electrode system is concentrated on metallic current collectors. Employing such collectors greatly simplifies fabrication and provides a mechanically strong system that can sustain high-current density. Metallic electrodes operate at a much lower temperature than ceramics, resulting in an increase in thermal flux and boundary-layer voltage drop. Both of these conditions lower the performance of an MHD generator. Also, because a cold system operates in the arcing mode, wastage of the electrodes must be carefully considered. The decrease in performance is not expected to be significant and the wastage of electrodes can be minimized by providing an appropriate cladding material. Candidate "cold" anodes and cathodes are being tested using a variety of oxidation-resistant claddings. Following completion of

specifications for the channel and extensive procurement activities, a contract was awarded for its manufacture. Diffuser, steam-generator adapter, and U-25 nozzle specifications were completed, and bids received and evaluated. Manufacture of a small channel for testing electrodes is progressing. A computer-based data acquisition system was purchased and installed. Software suitable for gathering, logging, and displaying data from various instruments has been developed. Related efforts included formulation of a plan for testing the U-25 generator in Moscow and making operational a one-dimensional finite-difference computer code for gas dynamics calculations. This code will be used during testing in Moscow. Although initially intended only for use during the testing of the U-25 generator, the system was recently shipped to the Soviet Union for the joint U.S.-U.S.S.R. experiments with the superconducting bypass magnet.

**PLANS FOR THE COMING YEAR** — Fabrication of the nozzle and diffuser/steam-generator adapter will be completed; the channel and instrumentation will near completion. It is anticipated that considerable effort will be directed toward finalizing the test plan and interfacing with the Soviets concerning the test program.

### **U.S. U-25B MAGNET (SCMS)**

**ARGONNE NATIONAL LABORATORY**

**DOE - \$1,074,000**

**6/75 - Continuing**

**OBJECTIVES** — The U.S. Superconducting Magnet System (SCMS) project involves analyzing, designing, fabricating, and delivering the SCMS to the bypass loop (U-25B) of the U-25 MHD facility in Moscow on a loan basis as agreed to in the U.S./U.S.S.R. Cooperative Program for MHD Power Generators, and subsequent joint testing of MHD channels in the jointly developed U-25B facility.

**RECENT WORK AND ACCOMPLISHMENTS** — ANL has designed, constructed, and tested a superconducting dipole magnet system consisting of the superconducting magnet, a helium refrigerator/liquifier installation, a helium gas-recovery system, and control equipment necessary to operate the magnet and its associated cryogenic equipment. The system was successfully tested to full design field in May 1977, in the United States. It was then safely delivered to Moscow in June 1977, and, in September 1977, cooled down and operated at the 5 T design field. The extensive effort involved in the fabrication of the magnet required close coordination of and good communication among the various participating organizations. Several divisions of ANL and several outside contractors contributed to this effort, all working to a stringent time schedule on a delicate first-of-a-kind product. Close teamwork was essential to successful completion of the construction phase. For economy, the coils use both high-field and low-field grade conductors. The joints between the lengths of conductor are made by a unique ultrasonic welding process designed and perfected for this particular application. To provide for efficient and accurate construction of the coils, a servomechanized winding system was developed. The mechanism consists of a turntable supported by a horizontal coil-winding drum. As the table rotates about its vertical axis, the cylinder is made to revolve about its horizontal axis with a rocking motion and the two motions are coupled by an electronic servo-network. The performance testing of the magnet system took place at ANL. The test program included evaluation of magnet operation, verification of compliance with the field specifications, determination of cryogenic operating parameters, and field mapping. The testing was accomplished with a partial cryogenic support system, because components of the

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system's refrigeration equipment had been shipped to the U.S.S.R. in July 1976, so that they could be installed and checked out prior to the arrival of the balance of the magnet system. The cooldown process of the magnet cryostat was accomplished in three phases, and very little trouble was encountered. Operation of the system indicates a total heat transfer to the helium of 5.5 w. To check out the energy dump system, the magnet was energized at incremental steps of current. The magnet was finally charged to full field without any problem on May 10, 1977. The field within the MHD warm bore was measured with a search coil, and the design field was achieved. The fringe field was also carefully mapped and documented. Shipment of the magnet system was a complicated activity that necessitated close coordination among representatives of ANL, DOE, the U.S. State Department, the U.S.M.A.C., and the U.S.S.R. State Department. The shipment began on June 18, 1977, when the magnet system left ANL, and was completed, via a nonstop flight on a U.S.M.A.C. C5 air-transport, in Moscow on June 20, 1977. The magnet system was accompanied to the U.S.S.R. by a team of ANL magnet specialists, who coordinated and supervised the installation and commissioning activities. The system installation was completed on schedule on July 4, 1977, and cooldown began. Cooldown proceeded well and paralleled the ANL experiences until early August of 1977, when severe difficulties were encountered with water in the Soviet gas supply/storage system. After a period of investigation and purification, the commissioning effort was renewed. The magnet was energized to the 5 T design field on September 29, 1977.

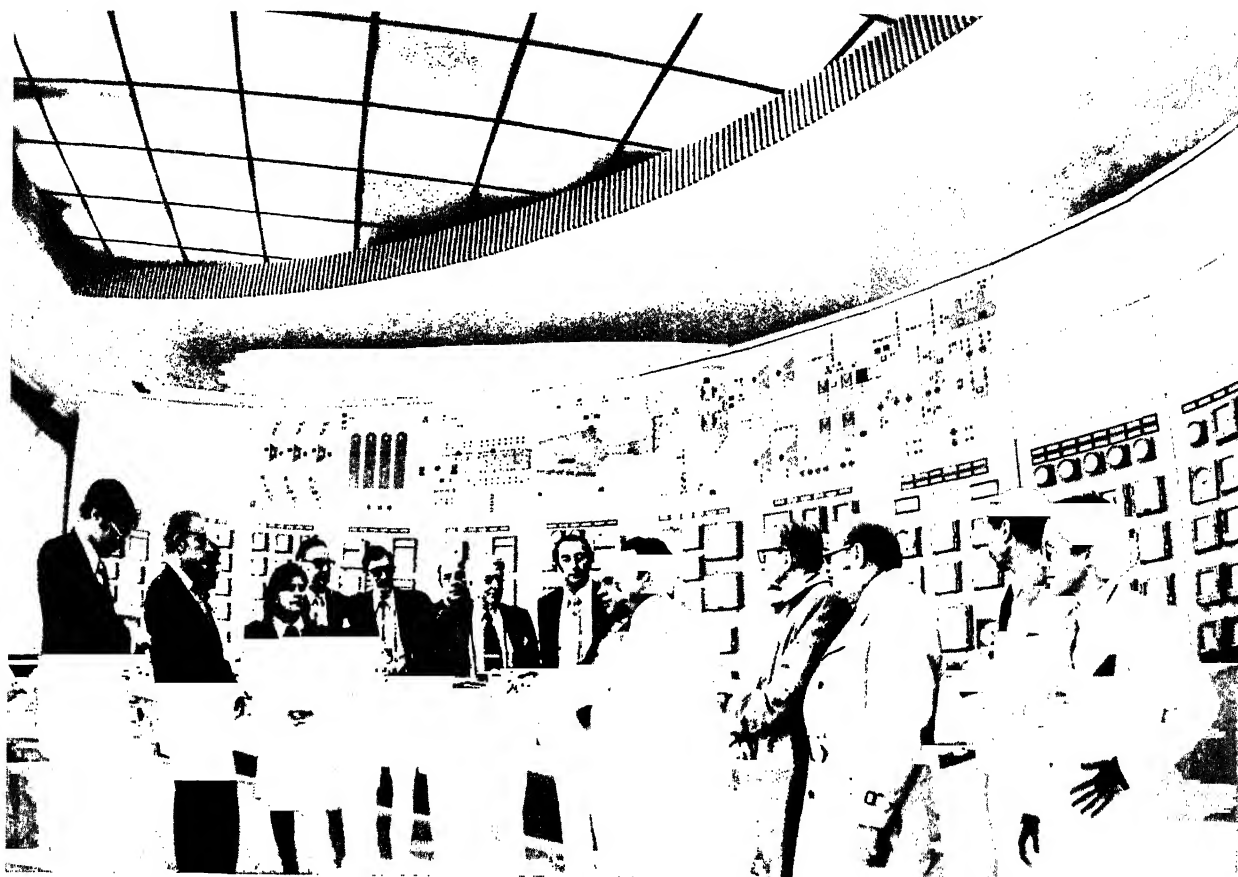
**PLANS FOR THE COMING YEAR** — ANL is responsible for maintaining the magnet system in the U.S.S.R. and for all subsequent operation of the system and phases of the joint generator test program to be carried out in the U-25B bypass loop. An ANL magnet specialist and a stress analyst will participate in all MHD channel tests employing the U.S. SCMS. The work will maintain and follow up mutually-agreed-to action items; conduct preparatory analytical studies to permit timely reduction of the data acquired; develop test plans jointly with the U.S.S.R.; participate in joint tests with the U.S.S.R. and monitor the condition of the magnet system before, during, and after testing; analyze and report on data acquired; and assure reliable operation and maintain the integrity of the magnet system. Testing of the magnet with a channel in an MHD generator mode has successfully taken place. Several subsequent tests are planned for 1978.

## **JOINT U.S./U.S.S.R. STATUS REPORT ON OPEN-CYCLE MHD ELECTRIC POWER GENERATION**

ARGONNE NATIONAL LABORATORY  
DOE - \$100,000  
6/75 - Continuing

**OBJECTIVES** — The joint U.S./U.S.S.R. status report on open-cycle MHD electric power generation is intended to summarize the state-of-the-art and to detail the requirements for successful commercial implementation of the process. By assembling in a consistent format the evaluations provided by the leading investigators in this specialized area of engineering, a unique handbook will result for use by specialists whose participation will be necessary for the advancement of MHD power generation and application. As the first publication of its kind, drawing upon the experimental and operational data of the two countries that are spearheading progress in MHD power generation, the joint status report can guide engineers and scientists of many disciplines into a technical area that holds promise of environmentally acceptable and economically feasible utilization of our extensive coal resources. Recent progress in design and development of the Component Development and Integration Facility and the Engineering Test

Facility, together with the upgrading of the Arnold Engineering and Development Center, have added greatly to the significance and usefulness of this MHD technology report.



*Contributors to Joint Status Report and Members of U.S. Staff for International Cooperative Program in MHD Toured U-25 MHD Pilot Plant at Institute of High Temperatures*

**RECENT WORK AND ACCOMPLISHMENTS** — The last in a series of intensive working sessions of the principal editors, the technical editors, and the American and Soviet authors was held in June and July of 1977 at ANL; technical editing is virtually complete. Earlier working sessions had been held in Moscow and the United States during 1976 and 1977. In the course of the work, the principal editors drew together a group of 29 outstanding technical authorities in the fields of MHD technology and research to serve, in addition to themselves, as chapter authors. This group, together with an additional 31 scientists and engineers whose contributions to various chapters are acknowledged by the authors, represents the lead organizations in MHD study and experimentation in the United States and the Soviet Union. The work of the chapter authors and contributors was coordinated on the Russian side through the Institute of High Temperatures of the U.S.S.R. Academy of Sciences, and on the American side through ANL. The American technical organizations represented include AVCO Everett Research Laboratory, Massachusetts Institute of Technology, NASA-Lewis Research Center, National Bureau of Standards, Pittsburgh Energy Research Center, Stanford Research Institute, and Tennessee Valley Authority. Without question, the liaison and correspondence generated through this extensive cooperation in the joint status report has given impetus to the development of MHD technology as an energy alternative.

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**PLANS FOR THE COMING YEAR** — The status report will be issued in hard-cover book format early in 1978. An assessment of the developing MHD technology will be sustained to assure timely issuance of updated chapters of this first status report. This publication is essentially an applied engineering report, which the principal editors believe is one in a series that will document the evolution of the technology and be extensively referenced. It is hoped, further, that the book can be used as a reference in colleges and universities with appropriate technical curricula. The book comprises a critical overview of the status of MHD technology at the time of publication, as well as of latest developments in the specialized fields of engineering that contribute most significantly to the evolution of this new form of energy conversion and resource conservation. Analyses of technical-economic technology aspects are included, with particular focus on commercial-scale open-cycle MHD electrical power stations and on step-by-step implementation of national programs for utilizing the unique benefits of an emerging energy alternative in behalf of the citizens of the two countries.

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Wright, R.J. "Coal-Fired Combustors for MHD Power Generation." Presented at the Fourth Annual International Conference on Coal Gasification Liquefaction and Conversion to Electricity, University of Pittsburgh, Pittsburgh, Pa., 2-4 Aug. 1977.

Wu, Y.C.L. "Performance Theory of Diagonal Conducting Wall MHD Generators." *American Institute of Aeronautics and Astronautics Journal* 14, No. 10: 1362-68 (Oct. 1976).

The following articles were presented at the Conference on High Temperature Sciences Related to Open-Cycle, Coal-Fired Magnetohydrodynamics Systems, Argonne National Laboratory, Argonne, Ill., 4-6 Apr. 1977 (ANL 77-21):

Anderson, J. et al. "Some Thermionic Emission Properties of Synthetic Coal Slag."

Bergman, P.D. "Seed Recovery - Conversion of  $\text{SO}^\ominus$  to  $\text{CO}_3^\ominus$ ."

Capps, W. "Coal Slag Properties Related to MHD."

Eliezer, N. et al. "Vapor Pressure Measurement Studies on Slag-Seed Component Equilibria."

Lu, C.L. et al. "Thermochemical Effects in the Determination of Electrical Conductivity in Coal-Fired MHD Plasmas."

Martin-Sanchez, M.; Kolb, C.E.; and Kenebrock, J.L. "Theoretical Analysis of the Effects of Slag Condensation on Plasma Conductivity in Open-Cycle, Coal-Fired MHD Generators."

Plante, E.R. "Some Vapor Pressure Data on  $\text{K}_2\text{O}$  Containing Substances."

Schneider, S.J. "Status of MHD Materials."

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Shen, L. et al. "Investigation of the Potential of Acid Washing of Coal Ash to Enhance Potassium Recovery In a High Ash Carry-Over Direct Coal-Fired MHD System."

Spencer, F.E., Jr.; Hendrie, J.C., Jr.; and Wildman, D.J. "Thermochemical Modeling of Combustion Plasma Mixtures."

The following articles appear in *Proceedings of the 16th Symposium on Engineering Aspects of Magnetohydrodynamics*, University of Pittsburgh, Pittsburgh, Pa., 16-18 May 1977 (CONF-770530):

Aspnes, J., and Pierre, D.A. "Dynamic Modeling and Control of MHD/Steam Electrical Power Generating Plants."

Bergman, P.D. et al. "Economic and Energy Considerations into MHD Seed Regeneration."

Bowen, H.K. et al. "Design and Performance of High Temperature Ceramic Electrode Modules."

Burenkov, D.K. et al. "Study of the Maximum Hall Voltages and Interelectrode Breakdown in the Channel of an Open Cycle MHD Generator: A Joint U.S. - U.S.S.R. Experiment on the UO2 Facility MHD Generator."

Capps, W. "Some Properties of Coal Slags of Importance to MHD."

Crawford, L.W. et al. "Generator Wall Slag Coating and Material Corrosion Experiments."

Cutting, J.D.; Maxwell, C.D.; and Ling, R.T. "Calculation of End Effects in Open-Cycle MHD Power Generators."

Doss, E. "Subsonic MHD-Diffuser Performance With High Blockage."

Fabris, G. et al. "Initial Generator Tests With Revised Ambient-Temperature Liquid Metal MHD Facility."

Frederikse, H.P.R., and Hosler, W.R. "Electrodes and Insulators; Design and Materials Considerations."

Lawit, R.L. "Open Cycle Coal Burning MHD Power Plants for Commercial Service."

—; Stoudt, R.A.; and Klett, M.G. "Status of the Reference Dual Cycle MHD-Steam Power Plant."

Loubsky, W.J. et al. "Molecular Gas Performance of a Disk Generator With Swirl."

Martinez-Sanchez, M.; Kolb, C.E.; and Kerrebrock, J.L. "Potential Effects of Coal Slag Condensation on Plasma Conductivity in MHD Generators."

Maxwell, C.D. et al. "Coupled Electrical and Fluid Calculations in the Cross Plane in Linear MHD Generators."



- Muehlhauser, J.W. et al. "Experimental Investigation of Multiple Loaded Diagonal Conducting Wall Generators."
- Pierson, E.S.; Dauzvardis, P.V.; and Dunn, P.F. "Sodium-Nitrogen Liquid-Metal MHD Facility Initial Test Results."
- Robles, T.; Johnson, R.; and Knox, J. "A Heat Capacitor for MHD Electrical Power Generation System."
- Rudins, G. et al. "The Second Joint Test of a U.S. Electrode System in the U.S.S.R. UO2 Facility."
- Scott, M.J., and Dicks, J.B. "Plasma Luminosity Fluctuations as a Diagnostic Tool."
- Tempelmeyer, K.E. et al. "Investigation of Factors Influencing Potassium Seed Recovery in a Direct Coal-Fired Generator System."
- Williams, J.E.C. et al. "Superconducting Magnet Development for the MHD Program."
- Wu, Y.C.L., and Rajagopal, G. "Three Dimensional Current Distribution in Diagonal Conducting Wall Channels."

The following articles appear in *Proceedings of the Sixth International Conference on Magnet Technology*, Bratislava, Czechoslovakia, 29 Aug. - 2 Sept. 1977 (CONF-770842):

- Wang, S.T. et al. "The U.S. SCMS Dipole Magnet System for the Bypass Loop of the U-25 Facility."
- . "Mechanical Perturbations Studies on Large MHD Superconducting Magnets."

The following articles appear in *Proceedings of the International Cryogenic Engineering Conference*, University of Colorado, Boulder, Colo., 2-5 Aug. 1977 (CONF-770801):

- Kim, S.H.; Wang, S.T.; and Turner, L.R. "Effects of Electrical Shorts on Cryostatic Stable Superconducting Magnets."
- Niemann, R.C. et al. "Cryogenic Aspects of the U.S. SCMS Superconducting Dipole Magnet for MHD Research."
- Wang, S.T. et al. "Fabrication Experiences and Operating Characteristics of the U.S. SCMS Superconducting Dipole Magnet for MHD Research."

The following articles appear in *Proceedings of the Liquid-Metal Power Generation Conference*, Rand Corporation, Washington, D.C., 19-20 Sept. 1977:

- Dunn, P.F. "Liquid Metal MHD Generators."
- Fabris, G. "Mixers and Surfactants."

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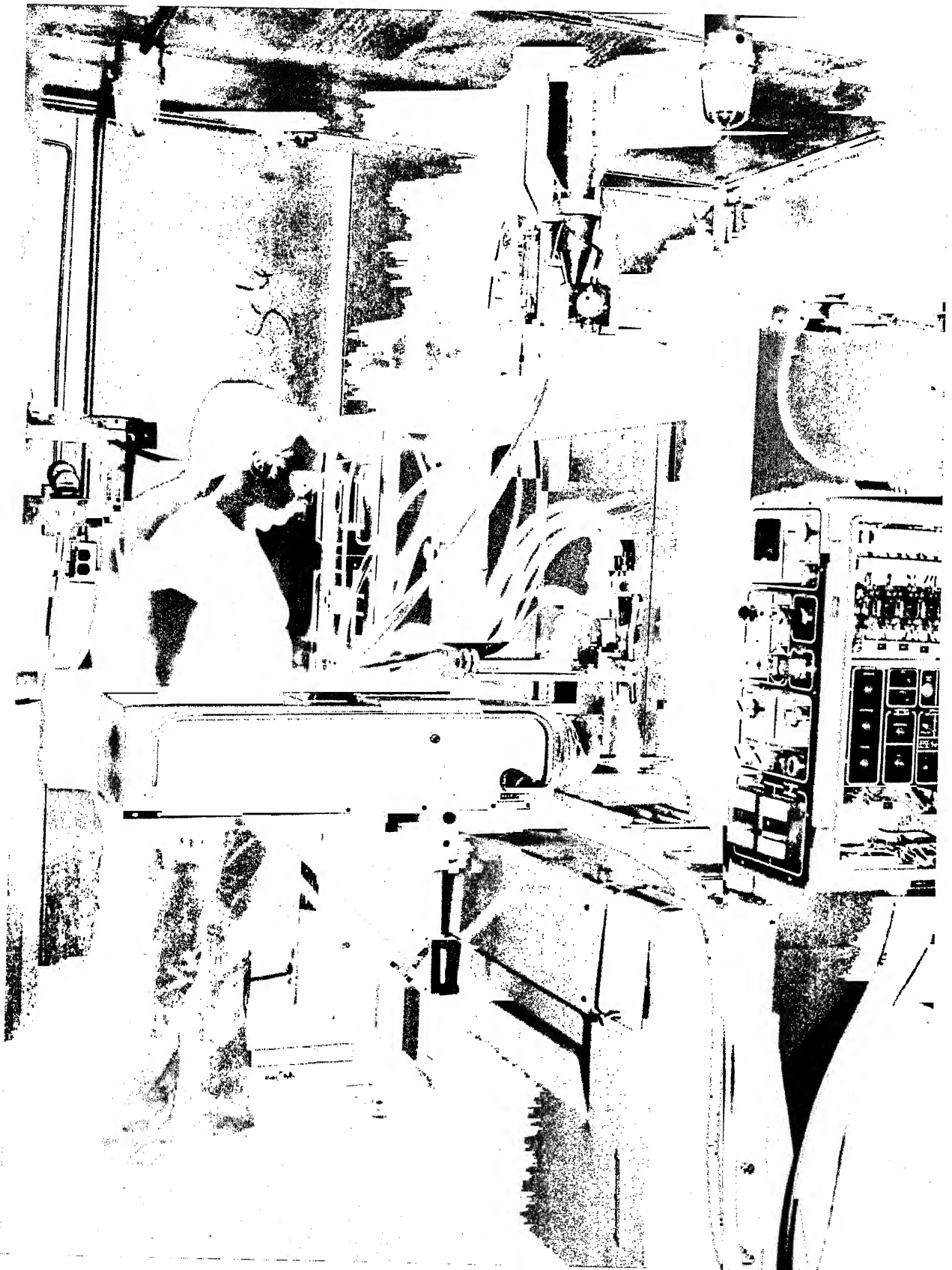
Petrack, M. "Rationale for Development of Liquid Metal MHD Power Systems."

—. "Summary Remarks."

Pierson, E.S. "Central Station Application of Brayton Cycle Liquid-Metal MHD."

—. "Development Plan at ANL."

Reed, C.B. "Rankine-Cycle Liquid-Metal MHD."



*Flame-Spraying Raney-Nickel Catalyst (Used in Methanation Reactor)*

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# ***ADVANCED RESEARCH AND SUPPORTING TECHNOLOGY***

The successful implementation of the Department of Energy's accelerated Fossil Energy Program requires that the research, development, and demonstration aspects of the program be pursued vigorously. The program was structured to assure both the timely accomplishment of necessary advanced research and the development of appropriate support technology to meet the Department's goals relative to fossil fuels. In particular, accomplishment of the AR&ST program will result in the establishment of second- and third-generation improved capabilities to utilize fossil fuels directly as energy sources or to convert these fuels to synthetic fuels. Serving as the research focal point for all elements of fossil energy, the AR&ST's research program is organized to implement the following objectives:

- Develop innovative fossil energy technology leading to significantly cheaper synthetic fuel processes for: gasification, liquefaction, and refining and chemicals
- Improve the operational reliability and efficiency of synthetic fuel plants through research on more corrosion-erosion-resistant materials and components
- Develop advanced techniques to permit increased combustion of coal in an environmentally acceptable manner
- Assure a supply of technically trained personnel from the Nation's university system.

Advanced Research and Supporting Technology is distributed among three major categories into which the work falls: Processes (coal conversion), Direct Utilization (coal combustion), and Materials and Components. Promising results during the past year from this advanced research include:

- A new, more economical, catalytic coal gasification process
- Improved yields of liquids and gases by flash hydropyrolysis of coal
- More selective catalysts for coal liquefaction
- Unusually high-octane gasoline from coal-derived methanol
- Demonstration of catalysts that are highly active and selective in refining coal and oil shale liquids
- A process for "chemical beneficiation" of coal that removes all pyritic sulfur and considerable organic sulfur
- Improved coal combustion techniques to decrease  $\text{NO}_x$ .

Likewise, significant progress was made in supporting technology, such as establishment of: more quantitative engineering of fluid-bed engineering; chemical structure of coal; mechanism of coal liquefaction; efficient, reliable valves for feeding coal to pressurized reactors; and accurate information on materials selection and failure prevention for coal gasification reactors.

## **PROCESSES**

The goal of AR&ST's liquefaction program is to develop advanced processes or uncover significant improvements in existing processes that will have technical, economic, and environmental advantages over current technology. In addition, it is an objective to carry out research that will lead to improved understanding of coal liquefaction. This work will lay the foundation for future technical and economic improvements in coal liquefaction processes. The major research categories are as follows:

- Extraction processes
  - Hydroextraction/desulfurization process research
  - CO-Steam process research
- Catalytic hydroliquefaction
  - Exploratory evaluation of catalysts
  - Slurry catalyst process
- Pyrolysis and indirect liquefaction
  - Flash hydropyrolysis
  - Indirect liquefaction from Syngas
- Refining and chemicals
  - Exploratory refining process
  - Refining of coal-derived syncrudes
- Supporting research
  - Basic chemical and engineering studies (e.g., structure of coal, preasphaltenes, mechanism of coal hydroliquefaction, thermodynamic properties of coal liquids, and catalytic reactor modeling/design studies).

Work is being carried out by industry, Energy Research Centers, National Laboratories, and others. Major accomplishments during the past year include:

A series of catalysts have been developed showing promise of superiority over conventional Fischer-Tropsch catalysts for converting synthesis gas to high-octane gasoline. Although there are still aging and regeneration problems, one of these catalysts converts 48 percent of a synthesis gas to a 93 research octane gasoline and another converts 77 percent to an 84 octane gasoline in short-term tests.

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- A  $\text{CO}^{13}\text{NMR}$  technique has been applied to coal to determine the functional groups present in solid coal. This work will be of considerable value in correlating the structure of coal with reactivity under liquefaction process conditions.
  - Experimental evidence was obtained indicating that inherent mineral matter in coal takes part in the conversion of preasphaltenes to asphaltenes.
  - A long-term operation (3790 hr) was obtained using a potassium-promoted Taconite catalyst for conversion of synthesis gas to oil (Fischer-Tropsch reaction). This catalyst can be used in a specially designed tube wall reactor that has the potential of significantly increasing the thermal efficiency of the Fischer-Tropsch reaction.
  - Results from noncatalyzed, short-residence time, rapid heatup hydropyrolysis tests at  $1300^{\circ}\text{F}$  have given carbon selectivities of distillate liquids of 30 to 35 percent. The liquid yields are significantly higher than those observed in other flash hydropyrolysis projects conducted at this temperature.
  - Coal liquefaction catalyst screening tests have indicated that cobalt/molybdenum on an alumina/zirconia support gives high coal conversion and selectivity to oil, compared to standard coal liquefaction catalysts. Further work is continuing to verify these results in a continuous flow unit.
  - Studies on the effects of mineral matter in coals have shown that pyrrhotite affects both coal conversion and oil yield while pyrite affects oil yield only. These results have provided insight into the role of mineral matter in the Solvent Refined Coal process.

Advanced research on coal gasification includes a continued search for more effective methanation catalysts, for processes involving the catalytic conversion of coal to hydrogen, and development of a catalytic coal gasification process. A major achievement in the latter area is the operation of a fluid-bed gasifier and evidence that the catalyst remains active for extended periods. Cleanup of gasification streams also has been an area of prime interest as has the further treatment of coal liquids to produce substitutes for refinery streams.

## DIRECT UTILIZATION

The objective of the AR&ST program in Direct Utilization is to advance the technology of direct utilization of coal. Exploratory research is focusing on coal beneficiation and feedstock control, combustion and energy conversion aimed at minimizing undesirable products ( $\text{SO}_2$ , particles,  $\text{NO}_x$ ), removal of undesirable components ( $\text{SO}_2$ , particles) from hot combustion products, and novel methods of heat transfer and power generation. The specific objective of the coal beneficiation research is improvement of economic and environmental aspects of coal combustion through precombustion treatment (quality monitoring, physical and chemical cleaning, comminution technology). Recent advances include laboratory demonstration and assessment of the chemical comminution potential and of the steam-air desulfurization. Combustion research is aimed at the advancement of combustion technologies that promote utilization of coal and/or suppress the formation of undesirable products. Among these technologies are fluid-bed combustion of coal, preparation and combustion of coal/oil slurries, and modifications minimizing the

formation of  $\text{NO}_x$  and of slag-forming species in pulverized-coal combustors. Recent advances include laboratory development of improved  $\text{SO}_2$  sorbents for fluid beds, and bench-scale development of tests for quantitative prediction of ash-fouling tendencies of various coals in actual boilers. Combustion chemistry and mechanisms of coal combustion have received increased emphasis as new, more precise analytical instrumentation has become available.

## **MATERIALS AND COMPONENTS**

Successful development of most coal conversion processes will depend on the ability of materials and components to withstand adverse operating conditions. Advanced coal conversion utilization processes are expected to operate at higher pressures and temperatures than conventional units to increase efficiency. For maximum economy, very large process units are also highly desirable. They require a strong materials and components development program to assure the reliability of future advanced coal conversion and utilization plants and to provide low-cost materials and components that simultaneously have a long service life. To achieve these goals, the following materials and components R&D program is being implemented:

- Exposure of materials coupons in pilot and demonstration plants
- Failure analysis of materials used in pilot plants
- Development and application of nondestructive testing
- R&D on corrosion, erosion, abrasion, and other forms of materials deterioration from coal handling, preparation, feed injection, gasification, and liquefaction.
- Development of ceramics to substantially improve the ceramic liners now in use
- Development of sulfidation-resistant alloys using non-critical raw materials
- Development of economical wear and abrasion-resistant alloys
- Development of pressure vessel alloys of high toughness strength and weldability
- Development of corrosion-resistant coatings
- Development of narrow gap field welding processes for thick-section pressure vessels
- Establishment of materials data information center and newsletter
- Development of lockhopper valves to perform reliably under the harsh environments of coal conversion processes

tion of high-pressure high-flow oxygen compressors

reliable, economic, high-pressure slurry coal feeding system for gasifiers

high-pressure high-temperature large-volume centrifugal oil slurry pump  
on plants.

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# **PROCESSES**

## **CO-STEAM/SLURRY CATALYST LIQUEFACTION**

**PITTSBURGH ENERGY RESEARCH CENTER**  
DOE - \$150,000  
1977 - Continuing  
Principal Investigators - H.R. Appell, E.G. Illig, F.W. Steffgen

**OBJECTIVES** – The objective of this work is to extend the scope of the Co-Steam process to high-sulfur bituminous coals. This objective was approached from two directions: to evaluate a variety of high rank coals to determine their reactivity in Co-Steam processing and to correlate their behavior with mineral content and other coal properties, and to improve the utilization of hydrogen in the synthesis gas by cheap, slurry catalysts that would increase the efficiency of the liquefaction process and reduce the high viscosity of the products generally obtained from bituminous coal and lignites. Co-Steam has the potential of becoming the lowest cost coal liquefaction process (low cost coal, low cost reducing gas, and no fixed bed catalyst).

**RECENT WORK AND ACCOMPLISHMENTS** – Various bituminous coals have shown a wide variation in reactivity towards Co-Steam processing. The ash content, especially the pyritic sulfur level, of the coal has a major influence on reactivity. The organic structure (e.g., coal rank) also seems to be quite important. An evaluation of bituminous coals for Co-Steam processing uncovered four that were actually more amenable to this low-cost method of processing than the North Dakota lignites for which the process was originally developed. The common characteristics of these highly reactive coals were a significantly higher than normal pyrite content, and a rank of hvCb or hvBb. These coals are prime candidates for liquefaction because their sulfur content is too high for direct combustion without stack gas scrubbing to remove sulfur dioxide, and their activity is so high that they can be liquefied in a self-catalytic process to yield low-sulfur, low viscosity products. The search for new catalyst systems for liquefying coal led to the combination of lead sulfide and ammonium chloride and/or hydrogen chloride. This new combination showed unusually high activity in converting coal to low viscosity liquids containing appreciable contents of benzene, toluene, xylenes, and also a naphtha fraction. Unlike earlier highly reactive catalysts (e.g., tin, iodine), the lead sulfide plus halide promoter is a low-cost catalyst. An advantage of this system over zinc chloride is that catalytic amounts instead of massive amounts can be used to obtain extensive liquefaction.

**PLANS FOR THE COMING YEAR** – The scope of Co-Steam processing will be enlarged and defined in terms of processibility of various coals. The effect of petrographic composition and the role of indigenous mineral matter will be correlated with the reactivity of coals. The use of low-cost disposable mineral catalysts will be further explored to increase reaction rates and extend the scope of Co-Steam to include many bituminous coals that cannot be efficiently processed under Co-Steam conditions in the absence of catalysts.

## **EFFECT OF TUBULAR PACKING ON COAL LIQUEFACTION PROCESSING**

**MORGANTOWN ENERGY RESEARCH CENTER**  
DOE - \$400,000  
7/74 - Continuing

**OBJECTIVES** – Coal liquefaction research is being conducted at MERC to explore and obtain basic information on the effect of non-catalytic packing material on the hydroliquefaction of coal to a high quality fuel oil.



**RECENT WORK AND ACCOMPLISHMENTS** – Laboratory-scale coal hydroliquefaction experiments were conducted to determine the effect of different types of nominally noncatalytic packings on the reactions of a coal/hydrogenated vehicle oil slurry in a first-step tubular reactor. This work included 20 runs at short residence time (15 minutes at temperature) made in a 1-gallon stirred batch reactor. Tests with Kentucky No. 9 coal showed that when packing was present only 10.3 weight percent of the undesirable asphaltenes were formed in the liquid product from the catalytic second stage compared to 14.5 weight percent when no packing was used in the first stage. Tests with Arkwright coal slurries showed 81 percent coal conversion using zirconia packing and 80 percent using alpha alumina packing, compared to only 75 percent conversion when no packing was used. Scanning electron microscopy and x-ray diffraction analysis of the used packings showed retention of appreciable amounts of mineral matter from coal on their surface, including pyrrhotite that resulted from the reduction of pyrite. These minerals, especially pyrrhotite, may be responsible for the increased reactions in the presence of packing as compared to no packing. Additional data showed that the coal minerals were responsible for a higher atomic H/C and a higher hydroaromatic (hydrogen donor) content for the product oil, asphaltenes, and residue (benzene insolubles) when the unfiltered first-step product was used in the hydrocatalytic reactor. It was also shown that the order of increasing hydrogen donor activity of the hydroaromatics was tetralin, hydrophenanthrenes, and hydrophenyrenes; that is, the 2-, 3-, and 4-ring compounds, respectively.

**PLANS FOR THE COMING YEAR** – During FY 1978, the research on hydroliquefaction of coal will include a study of catalyst deactivation by organometallics present in coal, the objective being to prolong the life of the hydrogenolysis-heteroatom removal catalyst by removal from the coal liquefaction reaction mixture of catalyst deactivating organometallics that are soluble in the product oil.

## UTILITY OF HYDROGEN-PERMEABLE CATALYTIC TUBULAR REACTORS

MORGANTOWN ENERGY RESEARCH CENTER  
DOE - \$150,000  
7/76 - 9/77

**OBJECTIVES** – A novel catalytic coal hydrogenation system was devised and studied for its conversion efficiency at low pressure and for catalyst durability.

**RECENT WORK AND ACCOMPLISHMENTS** – Exploratory research was conducted on a system consisting of hydrogen permeable catalyst reactors made of thin-wall nickel tubing, porous nickel-molybdenum tubing, and porous cobalt-molybdenum tubing. Each tubing was coated on the inside with a layer of catalytic metal sulfide by reaction with hydrogen sulfide or an organic sulfur compound. Hydrogen for the reaction diffused under differential pressure through the tubing wall and the catalytic coating. Feeds for the reaction consisted of coal tar oils and also pure compounds, such as phenanthrene and benzothiophene, as models for coal hydroliquefaction reactions. A series of runs with model compounds feeds, in thin-wall nickel tubing with an inner catalytic surface of nickel subsulfide, showed that most of the hydrogen, diffusing through the wall at 400° to 450°C and at a differential pressure of 700 to 750 psig, was consumed at an inner tube pressure of 1000 psig, to give up to 50 percent hydrogenation of phenanthrene and 56 percent hydrogenation of benzothiophene at residence times of 118 minutes. A series of runs with model compound feeds and coal tar creosote oil feeds was made in porous nickel molybdenum and porous cobalt-molybdenum tubing reactors. With the Ni-Mo tubing at 400°C, differential pressure 4.5 psig, and an

inner tube pressure of only 650 psig, there was 45 percent conversion of phenanthrene and 27 percent conversion of benzothiophene at the very short residence time of 3 minutes. The Ni-Mo tubing was therefore much more active than the thin-wall Ni tubing. With the Co-Mo tubing at only 350°C, differential pressure 20 psig, and an inner tube pressure of 750 psig, there was a minimum of 57 percent conversion of phenanthrene at a residence time of 11 minutes.

**PLANS FOR THE COMING YEAR** — Research on this project has been concluded. A manuscript summarizing the work has been prepared, reviewed, and approved for presentation and publication. In addition, the draft of a patent application has been prepared by the Oak Ridge Patent Group.

## NOVEL COAL/SOLVENT/HYDROGEN CONTACTORS

OAK RIDGE NATIONAL LABORATORY

DOE - \$140,000

7/1/76 - 9/30/77

**OBJECTIVES** — This work is designed to identify a feasible reactor for hydrogenating slurries of coal in a donor solvent under highly turbulent plug flow conditions and to obtain sufficient hydrodynamic data to compare different reactor configurations. Enhancement of reaction rates by using such a reactor might allow adequate hydrodesulfurization and liquefaction without the use of hydrotreating catalysts. There are a number of advantages to noncatalytic processes; namely, the cost of initial catalyst loading and the cost of periodic replacement of the catalyst are avoided. Enhancement of reaction rates by including small-scale turbulence could result in lower reactor operating temperatures, an important consideration for vessels operating at high pressure. A highly turbulent plug-flow reactor would be thermally more stable than a less turbulent reactor because of better heat transfer, and would give more efficient use of reactor volume than the ebullating-bed or bubbling reactors.

**RECENT WORK AND ACCOMPLISHMENTS** — Four reactor configurations that potentially could provide the necessary turbulence and plug flow conditions were investigated: packed columns, a column containing a Kenics in-line mixer, small-diameter open tubes, and a series of venturi contactors. With the first three configurations, axial mixing, gas and slurry holdups, pressure drop, and specific energy dissipation were measured over a range of gas and slurry (35 wt percent coal in water or glycerol solution) flow rates. A literature search and a very preliminary cost analysis indicated that the fourth configuration, consisting of many large venturis in series, would probably be impractical.

Hydrogenation of coal/solvent slurries in packed beds could provide the necessary turbulence and plug flow conditions and thus ought to serve as a baseline with which to compare other reactors. Increases in slurry flow rate are more effective at increasing energy dissipation (thus enhancing reaction rates) than increases in gas flow rate. Extensive measurements of dispersion coefficients show essentially plug flow behavior in the packed bed under the prevailing flow conditions. Data from the in-line mixer and the small-diameter open tubes show that slurry flow rates must be much greater than those through the same diameter packed bed to achieve the same energy dissipation. The small diameter open tube reactors also deviate significantly from plug flow.

**PLANS FOR THE COMING YEAR** — With the remaining funds, a few experiments will be performed in the coming year by the MIT School of Chemical Engineering Practice using the existing

experimental apparatus. These experiments will focus upon the effect of liquid property variations and upon additional contactor configurations.

## **DEVELOPMENT OF IMPROVED CATALYST FOR COAL LIQUEFACTION**

**PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$250,000**

**1976 - Continuing**

**Principal Investigators - R.E. Tischer, H.R. Appell**

**OBJECTIVES** – This work involves the development of new and improved catalysts for coal liquefaction that have acceptable life, high desulfurization and denitrogenation activity, and good selectivity for making stable, distillable liquids. Existing catalysts being used for coal liquefaction were tailored for processing petroleum and consequently are not resistant to the rapid coking and deactivation caused by coal components and do not have an active life satisfactory for commercial coal liquefaction.

**RECENT WORK AND ACCOMPLISHMENTS** – Work was initiated on the preparation of catalysts specifically designed for coal liquefaction. Lanthanum was shown to be a stabilizer for the gamma-alumina structure at low concentrations and to promote the formation of an amorphous structure at higher concentrations. Starting with a high-purity alumina having a larger than average pore diameter and incorporating cobalt and molybdenum oxides gave a catalyst showing significantly higher activity than a standard reference catalyst used in coal liquefaction. Testing procedures are inadequate for giving unequivocal differences between the high-activity catalysts being prepared and evaluated. In developing a new testing procedure, the various criteria being measured were evaluated. Conversion, product viscosity, and sulfur removal were shown to be most indicative of catalyst performance. These criteria are being considered in a new screening test for coal liquefaction catalysts that will be more sensitive to catalyst differences than earlier procedures.

**PLANS FOR THE COMING YEAR** – Development work on the new testing procedure will be completed and the test put into use. A series of catalysts containing combinations of cobalt, nickel, molybdenum, and tungsten sulfides on alumina-based supports of varying pore structure will be prepared and evaluated. After screening, the most promising catalysts will be used for additional evaluation in flow units under realistic processing conditions.

## **CHEMICAL CHARACTERIZATION, HANDLING, AND REFINING OF SRC**

**AIR PRODUCTS & CHEMICALS, INC.**

**DOE - \$640,258**

**6/20/75 - 10/1/77**

system includes two long-path reactors and one backmixed reactor designed for pressures up to 5000 psi and temperatures to 1000°F.

As a result of the handling study, it was concluded that very little, if any, change in chemical composition or heating value occurs upon storage of SRC; and the consolidation of SRC in storage bins and flowability of this material is highly dependent on the moisture content along with bin design. A pneumatic conveying system for SRC was designed and studied experimentally. The chemical properties of SRC have been evaluated. Samples from Wilsonville, Alabama, and Tacoma, Washington, have been studied. The carbon, hydrogen, nitrogen, and oxygen contents of these two independent SRCs are similar, with only the sulfur and ash contents slightly varying. The moisture content of the SRC was a function of the type of process used to form the solid.

SRC-containing filtrate has been successfully processed, and data from a continuous, two-phase, upflow reactor containing a fixed bed of catalyst have been evaluated and the following conclusions drawn: at typically commercial operating conditions of pressure, temperature, and liquid space velocities, greater than 90 percent desulfurization of SRC-containing filtrate has been achieved over a Co/Mo HDS catalyst; under similar conditions, nitrogen removal has approached 40 percent, and at these higher removals, noticeable hydrocracking took place. These experimental studies demonstrate that up to 16 percent ash, based on catalyst weight, can be successfully processed through the fixed-bed reactor without any aging effect on the catalyst, and comparative runs show NiMo on alumina is a better desulfurization and denitrogenation catalyst than NiW on silica alumina.

**PLANS FOR THE COMING YEAR** – Work on this contract has been completed.

## PRODUCING LIQUIDS BY SHORT-RESIDENCE-TIME COAL HYDROLYSIS

CITIES SERVICE RESEARCH AND DEVELOPMENT COMPANY

DOE - \$287,000

7/1/77 - 10/31/78

Principal Investigator - M.I. Green

**OBJECTIVES** – Results of experiments performed by Cities Service Research and Development Company (CSR), Brookhaven National Laboratories (BNL), City University of New York (CUNY), Institute of Gas Technology (IGT), and Pittsburgh Energy Research Center (PERC) have indicated that the noncatalytic, short-residence-time hydrolysis of coal is a potentially viable process for producing substitute natural gas (SNG) and attractive byproduct yields of mono- and di-aromatic liquids. With the recent increased demand for clean liquid fuels, there is an incentive for producing a slate of products consisting primarily of BTX, light oils, and heavier oils with lesser amounts of gas. CSR's bench-scale unit will be used to develop an understanding of the mechanism of short-residence-time hydrolysis; define the chemistry of BTX production from polyaromatic structures; determine operating conditions for maximizing the production of low-tar-containing, hydrocarbon liquids by noncatalytic, short-residence-time hydrolysis; define a standard liquefaction activity test (SLAT) for minimizing high test costs that would otherwise be required for screening catalysts and pretreatment methods; determine efficacy of coal pretreatment procedures on the short-residence-time hydrolysis process; determine efficacy of utilizing catalysts or pyrolysis modifiers in the short-residence-time hydrolysis process; and determine accurate values of coal particle residence times.

**RECENT WORK AND ACCOMPLISHMENTS** – A screening program was formulated to investigate the operability of several coal types in the bench-scale unit under short-residence hydrolysis conditions that favor maximization of liquids products. Several bituminous, subbituminous, and lignite coals were processed at a preselected, standard liquefaction activity test (SLAT) condition; SLAT defines a single set of coal and vapor residence times (about 3 seconds), reaction temperature (1300°F), rapid heatup rate (greater than 100,000°F/sec), reaction pressure (2200 psi), rapid quench temperature (about 700°F), and particle size distribution (plant grind). Operability of bench-scale hydrolysis reactors is a function of coal type and is controlled by the coking propensity of the aromatic oils derived from the initial, short-residence hydrolysis reactions. Agglomerating coals can be rendered non-caking without any substantial reduction in liquids yields during short-residence hydrolysis by low-temperature air drying.

**PLANS FOR THE COMING YEAR** – A process variable study will be performed to determine optimum conditions utilizing the candidate coal selected from the screening program. These conditions will be utilized to determine the effects on the overall hydrolysis process of applying specific catalysts or pyrolysis modifiers and decaking methods to coals prior to hydrolysis. Attempts will be made also to define the mechanism of BTX production from polyaromatic structures by studying the hydrolysis of model compounds.

## COAL PYROLYSIS BY HOT SOLIDS FROM A FLUIDIZED-BED COMBUSTOR

MASSACHUSETTS INSTITUTE OF TECHNOLOGY  
DOE - \$119,995; M.I.T. - \$10,000  
6/1/77 - 5/31/79

**OBJECTIVES** – This research is to determine the technical feasibility of generating clean, economically storable, liquid and/or gaseous fuels by the fluidized-bed pyrolysis of coal in the presence of lime and/or other inorganic solids capable of in-situ  $H_2S$  and  $CO_2$  removal. Specific objectives are to measure yields of gaseous, liquid, and solid products, gas compositions, and sulfur and nitrogen contents of chars and of successive distillation fractions of liquids from at least two coals of commercial importance. Pyrolysis is carried out in a fluidized-bed (2-in-I.D.) in the presence of lime under conditions of practical interest including bed temperature (800° to 1600°F), total external pressure (1 to 20 atm), coal feed rate (0.5 to 5 lb/hr), lime-to-coal ratio (0-50), coal and lime particle-size distributions (250 to 500 $\mu$ m), and fluidizing velocity (0.3 to 2 ft/sec). Related objectives are to develop global correlations and predictive models using these data. In the second year of the project, a preliminary engineering and economic assessment of the process will be performed by an industrial subcontractor.

**RECENT WORK AND ACCOMPLISHMENTS** – The design, construction, and testing of the fluidized-bed reactor and related product collection equipment has been completed: Material balances in the range 94 to 98 percent have been obtained with Montana lignite, a noncaking coal. Chromatographic methods for analysis of gaseous products as well as procedures for fractional distillation of small quantities of product liquids are under development. Other studies are focused on separation of char from mixtures containing sand and other solids used in the fluidized bed. All preliminary test runs with Illinois No. 6 bituminous coals have resulted in severe caking and subsequent plugups. Suitable operating conditions for handling such caking coals are being studied. The development of predictive models for the performance of the bed and eventual scale-up of the process is also underway.

**PLANS FOR THE COMING YEAR** – Systematic data collection will begin using Montana lignite as soon as acceptable protocols for gas analysis are completed. This will be followed by regular data collection using Illinois No. 6 bituminous coal, as soon as the determination of suitable operating conditions for handling caking coals is completed. The predictive models or performance equations under development will be tested and modified with the experimental data.

## DELAYED COKING OF COAL/PETROLEUM RESIDUAL SLURRIES

GULF RESEARCH & DEVELOPMENT COMPANY

DOE - \$2,439,500

6/76 - 10/80

**OBJECTIVES** – This program is designed to develop a near-term low-capital-cost coal liquefaction process using existing delayed coking capacity and conventional refinery technology to process coal/petroleum residual slurries and to upgrade the distillate product. This work is to be accomplished by developing a pyrolytic coking process where various coal/petroleum residuals are converted in delayed cokers to a coke distillate for fluid catalytic cracking to a high-octane gasoline or gasoline blending stock. The use of coal in this mode will also divert petroleum resid to fluid catalytic crackers for enhanced production of gasoline and fuel. This concept has the possibility of being implemented in today's refineries, since coal would be used to displace part of the petroleum residuals in existing delayed cokers, and could be commercialized in the 1980-1985 timeframe if successful. Assuming that 50 percent of U.S. delayed coking capacity is revamped to process coal/residual slurries and that the displaced residual oil is charged as an incremental portion of the feed to a fluid catalytic cracking unit, it is estimated that the increase in daily production of distillate and gasoline would be 90,000 and 70,000 bbl/d, respectively. The coke produced should be suitable either for power plant fuel or for gasification to hydrogen to meet refinery hydrogen requirements. As a fuel, the coke should be better than the parent coal since its ash and volatile content would be lower. Reduction of the volatiles would reduce the tendency to form soot, smoke, and tars on combustion of the coke as a power plant fuel.

**RECENT WORK AND ACCOMPLISHMENTS** – The product yield distribution obtained when coking various coal/petroleum residual slurries using both bituminous caking and subbituminous noncaking coals was investigated at normal coking pressure using a continuous slurry-feed heater and a 1-gallon coker. The results indicate that the synergism previously obtained in batch exploratory experiments is maintained when the slurries are heated rapidly to incipient coking temperatures using a continuous slurry preheater. Pressure coking of bituminous coal/residual slurries resulted in synergistic  $C_3+$  distillate yields up to 3.0 weight percent accompanied by an equal reduction in coke yield. The increase in  $C_3+$  distillate yield represents the net effect of a reduction in the naphtha yield, which is more than offset by the yield increases in the 221° to 343°C furnace oil and in the 343° to 566°C heavy gas oil. Product properties were also examined for the slurries and compared to linear predicted values. The naphtha and coke had lower than predicted sulfur while the furnace oil had more. All distillates had more saturates, hydrogen, and carbon, and less nitrogen, than predicted, indicating a synergistic improvement in product properties. The naphtha and furnace oil were less aromatic while the gas oil was more aromatic because of a substantial reduction in hexane insolubles. The lower than predicted carbon and higher than predicted ash, along with the predicted hydrogen content, in conjunction with a reduced coke yield, confirms that carbon and hydrogen are going to distillate, and less coke is being made. Generally, the synergism demonstrated by exploratory coking of the subbituminous non-caking coal slurries was somewhat greater than that seen with the bituminous caking coal slurries, with synergistic  $C_3+$  distillate yields of 6.5 weight percent.

Unlike other coal conversion processes using hydrogenated solvents, initial slurry viscosity measurements using a sand bath heated Brookfield viscosimeter showed only slight non-Newtonian behavior up to 370°C. This fact was confirmed by the lack of high back pressure during use of the continuous slurry heater for pressure coking and during slurry heater fouling studies. The critical decomposition temperature of 433° to 440°C was determined for bituminous and subbituminous/petroleum residual slurries at different slurry concentrations by employing an experimental fouling apparatus normally used for petroleum liquids. The effect of coal in reducing the allowable heat flux was demonstrated and preliminary evidence obtained indicating useful reaction of the coals and petroleum residual in the heater at temperatures between 400° and 435°C and residence times as low as 3 seconds. In laboratory-scale coking experiments with tagged compounds, it has been concluded that a high level of hydrogen transfer occurs during coking; however, much of this transfer is the result of hydrogen scrambling promoted by the reactor surface and/or coal mineral matter. There appears to be no transfer of carbon and essentially no donor solvent (such as that present in the residual) is retained in the coke or heavy liquids.

**PLANS FOR THE COMING YEAR** — Preliminary economic analyses of this process will be completed. Concurrently, plans will be prepared to expand the slurry heater studies to include the investigation of coal hydroliquefaction slurry heaters.

## SYNTHESIS OF LIQUIDS AND PIPELINE GAS BY A FISCHER-TROPSCH TYPE PROCESS

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$200,000

7/1/74 - Continuing

Principal Investigator - W.P. Haynes

**OBJECTIVES** — This project is aimed at developing design criteria and the data base for an advanced Fischer-Tropsch pilot plant. Research efforts include reactor development, optimization of operation conditions, and the determination of the best promoter to improve catalyst activity, life, and selectivity. The development of a co-products process to produce both synthetic liquid fuels and high-Btu gas by the Fischer-Tropsch reaction of coal-derived synthesis gas could significantly contribute to the initiation of a coal conversion industry.

**RECENT WORK AND ACCOMPLISHMENTS** — During the past year, efforts were concentrated on the development of a data base for flame-sprayed taconite catalyst. Synthesis gas conversion and product distribution have been measured at pressures of 300, 650, and 1000 psig; temperatures of 325° and 340°C; catalyst carburetion temperatures of 240° and 310°C; and with and without potassium impregnant. The best results to date have been obtained with a potassium-impregnated taconite that was carburetted at 240°C. This catalyst has been on stream for 4-1/2 months with no decline in activity. Maximum oil production has been realized at a temperature of 340°C and pressure of 650 psig. At these conditions, 89 percent of the total liquid hydrocarbon product falls in the gasoline range.

**PLANS FOR THE COMING YEAR** — The regenerability of taconite catalyst will be studied. Preliminary results indicate that satisfactory catalytic activity can be achieved by a secondary reduction-carburetion treatment of deactivated catalyst; confirmation will be sought. Tests of a PDU to provide scaleup design criteria will be started. Tests will be conducted also in both internally and externally sprayed tube wall extended-surface reactor systems.



## SLURRY CATALYST HYDROLIQUEFACTION OF COAL TO A READILY REFINABLE SYNCRUDE

SUNTECH, INC.

DOE - \$1,456,000

5/7/76 - 11/1/79

Principal Investigator - A. Schneider

**OBJECTIVES** – An economically and technically viable process for the catalytic hydroliquefaction of coal to a readily refineable syncrude for production of gasoline is being sought. The first step of the process consists of deep catalytic hydrogenation of coal in a suitable solvent, employing a relatively low-cost catalyst, and achievement of H/C atomic ratios high enough to allow application of catalytic cracking, hydrocracking, etc., to the product of hydrogenation; however, since experience has shown that the required simultaneous, very substantial conversions of nitrogen and oxygen compounds into hydrocarbons do not occur under conditions feasible for hydroliquefaction of coal, the chemical removal of these nonhydrocarbons is to be developed as the second step in the desired process. While modern petroleum hydrogenation catalysts are probably adequate for hydroconverting coal asphaltenes to distillable oil and excellent for desulfurization and hydrogenation of aromatics, the inability of these catalysts to hydrodenitrogenate during hydroliquefaction of coal without excessive hydrogen consumption is a major obstacle. Experience from the catalytic hydrogenation of petroleum residua and shale oil indicates that a large portion of the nitrogen remaining after catalytic hydroliquefaction of coal will be basic and could be precipitated with HCl. The combination of hydrogenation and HCl-treating is a potentially low-cost way of achieving nitrogen levels low enough to allow the application of modern petroleum refining processes to the resulting coal-derived syncrude.

**RECENT WORK AND ACCOMPLISHMENTS** – At 425°C, 2500 psig and 1 to 2 hours of contacting in stirred autoclaves, hydroliquefaction experiments on Illinois No. 6 coal slurried in hydrogenated anthracene oil (H/C atomic ratio 1.1 - 1.2) in the presence of 10 to 20 wt percent, based on total charge, of an in-house prepared catalyst consisting of 1 percent CoO - 2 percent MoO<sub>3</sub>-on-bauxite gave conversions of about 90 wt percent MAF coal to gases and liquid. Under these conditions, 80 to 85 wt percent of the coal-derived liquids distilled below 1000°F. Per ton of dry coal, around 11 to 17 Mscf hydrogen were consumed, or, assuming 4 barrels of product per ton of coal, about 3 to 5 Mscf hydrogen per barrel of product. Nitrogen contents of the total liquid products, filtered from catalyst, ash, and unconverted coal at room temperature, decreased with increasing severity of hydroliquefaction conditions, but only around a 1/3 reduction in nitrogen of the total charge (0.64 percent N) was achieved under the most severe conditions of hydroliquefaction employed. The 1000°F distillate portions of the filtered liquid products formed no precipitates on dilution with hexane or on saturation with dry HCl; however, high-nitrogen precipitates formed on saturating the 1000°F distillates diluted 1:1 with hexane with HCl, giving less than 0.1 wt percent nitrogen in the raffinate stripped of hexane. Conditions of increasing severity during the hydroliquefaction experiments resulted in smaller amounts of HCl precipitate, such that under the most severe conditions used, the weight of precipitate amounted to around 1/3 the weight of coal originally charged.

Types and concentrations of polynuclear aromatics in the charge to catalytic cracking must be considered as well as the concentration of nitrogen. Mass spectrometric analysis of the distillate fraction in the solvent boiling range of the filtered liquid product from a hydroliquefaction experi-



ment of coal with hydrogenated anthracene oil showed considerable decreases in tri- and tetrapolynuclear aromatics and increases in mono- and di-aromatics as compared with the unhydrogenated anthracene oil. Not only is improved quality for catalytic cracking indicated, but also nondegradation of the solvent for recycle to the catalytic hydroliquefaction step.

**PLANS FOR THE COMING YEAR** – Catalytically hydroliquefied coal in the gas oil boiling range, after treatment with HCl to remove nitrogen, will be assessed as catalytic cracking charge stock in a bench-scale, fluid catalytic cracking unit. Continuous fixed-bed hydrotreating of the gas oil product of catalytic hydroliquefaction, both HCl-treated and untreated, will be carried out to make product for evaluation as catalytic cracking charge stock. The conversion to 1000°F distillate of 1000°F<sup>+</sup> residuum from distillation of catalytically hydroliquefied coal by recycling to the hydroliquefaction step will be examined. A process will be developed for thermally decomposing the HCl precipitate to recover HCl for recycle and nitrogen concentrate for the generation of hydrogen. The 1 percent CoO - 2 percent MoO-on-bauxite catalyst will be evaluated in coal hydroliquefaction in continuous operation either in an outside, rented bench-scale unit or in an internally constructed and operated unit.

## SCREENING OF CATALYSTS FOR CATALYTIC HYDROLIQUEFACTION

BATTELLE, COLUMBUS LABORATORIES

DOE - \$1,218,600

6/1/76 - 6/15/78

**OBJECTIVES** – The development of one or more catalysts with demonstrated performance characteristics superior to those presently used for catalytic coal liquefaction is the goal of this program. More specifically, the catalyst should allow conversions at lower hydrogen consumption and have stability during the hydrogenation of asphaltenes. Methods to achieve this goal involve screening of catalysts and evaluation of the best candidates in longer aging studies. It is not intended here merely to screen presently available catalysts but to custom design a superior catalyst using background knowledge and experimental data generated during the program. A catalyst that will allow the conversion of coal to usable liquid in high yield under relatively mild conditions and, at the same time, have a long lifetime would ultimately permit a substantial decrease in the time needed to bring coal liquefaction processes to commercialization. A cobalt molybdate preparation, desirable because of its high desulfurization activity and low gas generation, is favored presently.

**RECENT WORK AND ACCOMPLISHMENTS** – Eight tabletted catalysts have been evaluated in a heavy oil vehicle and 10 extruded catalysts in tetralin. Relative effectiveness of catalysts was indicated by conversion of Illinois No. 6 coal under hydrogen in an autoclave to liquid products soluble in benzene in 2-hr runs at 425°C at 4000 psig (heavy oil) or 2500 psig (tetralin runs) total pressure. Only relatively minor differences in total conversion were observed among the catalysts, all of which showed a modest improvement over thermal (uncatalyzed) runs; however, significant differences were observed in the oil-to-asphaltenes ratio with high ratios coinciding with most effective desulfurization of the liquid product and, as might be expected, with the highest H-to-C ratios. Extruded (1/16 in.) catalysts appear to be more active than the 1/8-in. tablets. The initial screening tests have served to identify several catalysts that show better short-term performance than the reference CoMo tablet. A continuous reactor designed largely in imitation of the Synthoil reactor at PERC has been constructed and used for limited testing of the reference CoMo catalyst. This reactor is capable of processing up to 5 lb/hr of coal slurry at up to 4000 psig pressure and

about 850°F with hydrogen recycle. Problems have been encountered directly relatable to the unreliability of components operating under these coal conversion conditions. Catalyst removed after use from the 5/16-in.-by-70-ft fixed bed has been examined for changes in physical properties and mineral deposits as a function of the sample's location in the beds. Among other conclusions it would appear that fluid penetrates the tablet only about 300 micrometers.

**PLANS FOR THE COMING YEAR** – Additional catalyst formulations will be prepared, incorporating modifications based on prior experience, and screened in the rapid-screening autoclave reactor. Selected catalysts will be evaluated in longer (100-hr) runs at operating conditions less severe than previously used (e.g., 3000 psig) in the continuous reactor in hope of achieving greater operational reliability. A thermal run will also be conducted in the continuous reactor using glass bead packing in lieu of catalyst to provide a baseline for comparative purposes.

## BASIC AND EXPLORATORY STUDIES IN HOMOGENEOUS CATALYTIC COAL LIQUEFACTION

SRI INTERNATIONAL  
DOE - \$594,588  
10/1/75 - 10/1/77

**OBJECTIVES** – The principles of coal liquetaction chemistry are being studied in terms of the chemical fundamentals governing the process. The program is broken into four tasks: I—use of simple alcohols and water as the source for hydrogen in coal conversion; II—chemical details of the H-donor process in various media; III—establishing the fundamental chemical limitations in coal conversion processes in terms of the thermochemical and kinetic constraints operative under conversion conditions; and IV—structure of coal and its reactions with aqueous sodium hypochlorite.

**RECENT WORK AND ACCOMPLISHMENTS** – Task I. It was established that hydrogen donation to coal from simple alcohols such as methanol and isopropanol could be catalyzed by the addition of basic materials, such as potassium hydroxide. The procedure in some cases yields liquids with low nitrogen and sulfur values. Model compound work showed that the conditions utilized cleaved ether linkages, and added hydrogen to polyaromatic systems such as anthracene. Later the same reaction conditions, with water and CO used in place of alcohol, and with a bituminous coal, yielded a product with substantial increases in hydrogen content.

Task II. It was found that the H-donor process with tetralin was inhibited by treatment of the coal with pyridine. The donation of hydrogen to coal by tetralin was studied at reaction times in the range 20 to 250 sec. at 400°C with a number of diluents, and it was found that nitrogen-containing materials severely suppressed the process. These results suggest that the coal contains an effective catalyst for H-donation, and that the catalyst is easily poisoned by nitrogen.

Task III. An extensive compilation of experimental and estimated thermodynamic data was prepared for molecules related to postulated coal structures, and these data incorporated into group additivity procedures for rapid estimation of these properties. Additionally, a prediction scheme was developed for the thermochemical properties of polycyclic resonance stabilized radicals, many of which are, or closely resemble, key radical fragments produced in the thermal-bond cleavage reactions of coal structures. Testing on this predictive scheme has begun by means of very-low-pressure pyrolysis measurements of the rate of 1-ethylnaphthalene decomposition.

Task IV. Previous work elsewhere with aqueous hypochlorite gave mostly benzene carboxylic acids and carbon dioxide, and resulted in new and dubious conclusions about coal structure. Research showed that this oxidation at 30°C can give products ranging from good yields of acids of molecular weights up to 1000 to the products indicated above. The production of high molecular weight acids is favored by repeated oxidations with small proportions of hypochlorite and a controlled pH of about 11. Recent work has shown that use of very finely divided coal is also essential. Under optimum conditions only the most reactive aromatic systems (condensed and phenolic) are attacked, leaving a soluble product that is a new and promising material for investigation of coal structure. The behaviors of coal extract, extracted coal, and SRC were compared.

**PLANS FOR THE COMING YEAR** – Tasks I and II will be continued. The use of water as a hydrogen source for coal conversion will be exploited. Reaction conditions will be screened to optimize the process, and inexpensive bases, such as lime, will be used. Alcohol/base observations will be investigated further, and incorporation of the favorable nitrogen removal capabilities of these systems into SRC recycle solvents will be studied. Study of the fundamentals of the H-donor process will continue, with an investigation of the exchange of hydrogen between different coal extracts. Further, an attempt will be made to establish the nature of the H-donor catalyst in coal in experiments in which the mineral matter in coal is used in model compound studies. Finally, an acid/base nature of asphaltenes will be investigated in terms of the H-donor abilities of both the acid and base fractions.

## **FUELS AND PETROCHEMICALS FROM SYNTHESIS GAS**

**PITTSBURGH ENERGY RESEARCH CENTER**

DOE - \$160,000

1974 - Continuing

Principal Investigators - R.A. Diffenbach, H.R. Appell, F.W. Steffgen

**OBJECTIVES** – This research is aimed at developing catalytic processes for conversion of synthesis gas into products such as C<sub>2</sub>–C<sub>4</sub> olefins for petrochemical feedstock, high-Btu gas supplements or LPG, aromatic high-octane gasoline, and diesel fuel. Projections of U.S. demand for C<sub>2</sub>–C<sub>4</sub> olefins during the next decade indicate that a twofold expansion in manufacturing capacity will be necessary. Little work has been done on developing new catalysts for conversion of synthesis gas to hydrocarbons. Development and wide use of new types of catalysts (such as zeolites, bimetallic clusters), as well as significant improvements in catalyst characterization, make it appear likely that new catalysts can be developed with improved activity and selectivity.

**RECENT WORK AND ACCOMPLISHMENTS** – A catalyst screening program is presently under way using bench-scale gradientless (Berty) reactors to assure that temperature and mass transfer

**PLANS FOR THE COMING YEAR** – The catalyst screening program will continue. Three types of catalysts will be studied. These include Raney catalysts, multimetallic catalysts, and zeolites. The multimetallic catalysts will include alloys, clusters, or both. The metals Ni, Co, Fe, Ru, Cu, Pt, Ir, Be, Ag, Sn, and possibly others will be used in the multimetallic study. Zeolites and other oxide-type structures will be used to develop a process for converting methanol to  $C_2$ – $C_4$  hydrocarbons.

### COAL LIQUIDS UPGRADING

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$250,000

1976 - Continuing

Principal Investigators – Y.C. Fu, F. W. Steffgen

**OBJECTIVES** – This work focuses on developing and demonstrating techniques for upgrading coal-derived liquids to produce distillate fuels that will meet present and future fuel specifications. Combinations of refining processes that are suitable for coal liquids will be investigated.

**RECENT WORK AND ACCOMPLISHMENTS** – The building to house the small pilot plant units has been constructed, and the new facility was occupied at the end of the fiscal year. The laboratory will have two continuous reactors available to this project, a vacuum stripping unit, an oil storage and delivery system, and the necessary product handling facilities. Work is underway on autoclave experiments to compare converting coal liquids by single- and two-stage hydrotreating-hydrocracking. Factorial experiments of single- and two-step hydroprocessing with three variables of temperature, pressure, and time at three levels have been conducted. The feedstock selected as being representative of coal liquids for the factorial study is a mixture of Solvent Refined Coal and SRC solvent in a 30:70 weight ratio. Single-stage hydroprocessing of SRC blend over nickel tungsten catalyst did not achieve the degree of molecular weight reduction (conversion of pentane insolubles) or nitrogen removal necessary to produce high yields of clean distillate products. Two-stage hydroprocessing, in which the first-stage hydrotreating is conducted over a nickel molybdenum catalyst to prepare a feed for a second-stage hydrocracking using nickel tungsten catalyst, shows more conversion and, therefore, more promise. The sulfur and nitrogen contents of the second-stage turbine oil fraction would meet EPA standards. Regression analysis of data from the factorial experiments has provided quadratic expressions relating dependent variables (sulfur and nitrogen reduction, hydrogen consumption, gas make, and conversion of pentane insolubles) with the process variables (temperature, pressure, and residence time). From these equations, it is possible to determine what conditions will give a maximum conversion level of one dependent variable (e.g., nitrogen) without exceeding a desired level of another, such as hydrogen consumption.

**PLANS FOR THE COMING YEAR** – Work will be devoted to preparation and testing of new catalysts and to process combination studies to be conducted in bench-scale flow units. Based on the results, further process development and optimization studies will be made.

## CHARACTERIZATION OF SYNCRUDES FROM COAL

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$150,000

7/1/74 - Continuing

Principal Investigator - J.E. Dooley

**OBJECTIVES** – Liquids produced by coal liquefaction processes must be further upgraded to finished products in order to be used more effectively in energy systems. The basic compositional data needed for these refinements are not readily available. The objective of this project is to provide the appropriate compositional data for these coal liquids through a systematic characterization procedure, and thereby provide the basis for developing processes to upgrade the materials to more useful fuels. Although unique characterization techniques are already available at BERC for this task, other analytical methods will also be evaluated, modified, and developed as necessary to provide better capability for characterizing coal liquids.

**RECENT WORK AND ACCOMPLISHMENTS** – Characterization studies were completed for the  $>200^{\circ}\text{C}$  material from a coal liquid prepared by CONOCO using their  $\text{ZnCl}_2$  process for liquefying Colstrip coal. Work was initiated on six coal liquids prepared in a small batch autoclave. Most of the necessary separations on these six coal liquids were completed and mass spectral analysis of fractions are underway. Two more coal liquids will be included in this program, which will provide the first study that relates coal rank with the hydrocarbon structures that can be obtained from a given rank of coal.

**PLANS FOR THE COMING YEAR** – The characterization study for autoclave prepared coal liquids will be completed.

## METHANOL-TO-GASOLINE AND SASOL/FISCHER-TROPSCH TECHNOLOGY ASSESSMENT

MOBIL RESEARCH AND DEVELOPMENT CORPORATION

DOE - \$214,313

12/20/76 - 12/20/77

**OBJECTIVES** – A technical and economic comparison is being made between the Mobil methanol-to-gasoline technology, currently under development, with the commercial Fischer-Tropsch technology for the production of gasoline from Western coal. The co-production of SNG and other liquid hydrocarbons is taken into consideration. Two base cases are developed specifically for the Mobil fixed-bed and the Sasol-type, fluid-bed Fischer-Tropsch processes. The synthesis gas is produced by the commercial Lurgi dry-ash gasifier technology. In addition, sensitivity cases are developed for: producing fuel methanol and SNG (elimination of the Mobil technology); reforming the methane produced for maximum gasoline yield (elimination of the SNG co-product); substituting the Mobil fluid-bed process; substituting the noncommercial Winkler pressure, fluid-bed gasifier, under development; and using the Mobil direct-route technology, also under development, to produce gasoline directly from the synthesis gas in place of the Fischer-Tropsch technology. This venture study identifies the technical and economic feasibility of the Mobil methanol conversion technology and provides a firm guidance basis for future laboratory and pilot-plant programs. Moreover, this study can be easily adapted for continuing sensitivity cases to evaluate technological developments, not only in the Mobil technology but also in the gasification, synthesis gas purification, and alcohol synthesis technologies.

**RECENT WORK AND ACCOMPLISHMENTS** – Conceptual plant complexes have been developed for each of the Mobil and Fischer-Tropsch base cases. The complexes are self-supporting grass-roots facilities assumed to be located in a Wyoming coal field. Plant size is equivalent to the proposed 280 MMscf/d SNG plants. Except for the Mobil methanol conversion process and the developing pressurized Winkler gasifier technology, all processes used in the study are commercially available. The complexes have been designed to meet environmental regulations in Wyoming. Sufficient processing technologies or steps are included to upgrade all products to meet U.S. market specifications and to produce an SNG that is interchangeable with natural gas. A summary yield comparison between the two base cases is as follows:

|   | Base Case I: Mobil<br>Methanol Conversion | Base Case II:<br>Fischer-Tropsch |
|---|---|----------------------------------|
| Coal (as received) - t/SD                             | 27,334                                    | 27,792                           |
| Gasoline Produced - bbl/SD                            | 22,045                                    | 13,580                           |
| Substitute Natural Gas - MMscf/SD                     | 148.5                                     | 173.3                            |
| Other Fuels - bbl                                     | 3,760                                     | 4,182                            |
| Mixed Alcohols - bbl                                  | —   | 1,825                            |
| Total Product Output - FOE bbl/SD <sup>1</sup>        | 45,550                                    | 44,940                           |
| Percent Gasoline Produced (all products) <sup>2</sup> | 41  | 25                               |
| Thermal Efficiency (HHV), Percent                     | 62  | 58                               |
| Processing Steps after Gasification <sup>3</sup>      | 9   | 18                               |

<sup>1</sup> Each product is converted to its fuel oil equivalent (FOE) at 6.0 MMBtu/bbl.

<sup>2</sup> As percent of total heating value of all products.

<sup>3</sup> Additionally, there are nine gasification, gas purification, and byproduct recovery steps common to both cases.

The Mobil technology produces a greater quantity of gasoline (with no diesel, heavy fuels, or alcohol) at a somewhat higher overall thermal efficiency than the Fischer-Tropsch technology. Moreover, the percentage of SNG in the total produce is significantly lower when using the Mobil process. The product ratios are summarized below:

|                                    | Base Case I: Mobil<br>Methanol Conversion | Base Case II:<br>Fischer-Tropsch |
|------------------------------------|---|----------------------------------|
| 10 RVP Gasoline                    | 1.62                                      | 1                                |
| Gasoline and Heavier Fuels         | 1.34                                      | 1                                |
| Propane and Butane                 | 3.0                                       | 1                                |
| SNG                                | 0.86                                      | 1                                |
| Liquid Fuel/SNG Ratio <sup>1</sup> | 47/53                                     | 34/66                            |

<sup>1</sup> Based on thermal value (HHV) of products.

The Fischer-Tropsch process also yields a small amount of organic acids, which must be removed. Conversely, the Mobil process yields only trace amounts of organic acids. Along with its improved yield selectivity, the Mobil technology achieves marketable products via a considerable reduction in the number and complexity of required processing technologies or steps.

**PLANS FOR THE COMING YEAR** – The technical development is completed for the two base cases and the five sensitivity cases. The capital investment estimates and the economic evaluation are scheduled to be completed by December 20, 1977. The final report will be issued in early 1978.

### SEPARATIONS TECHNOLOGY

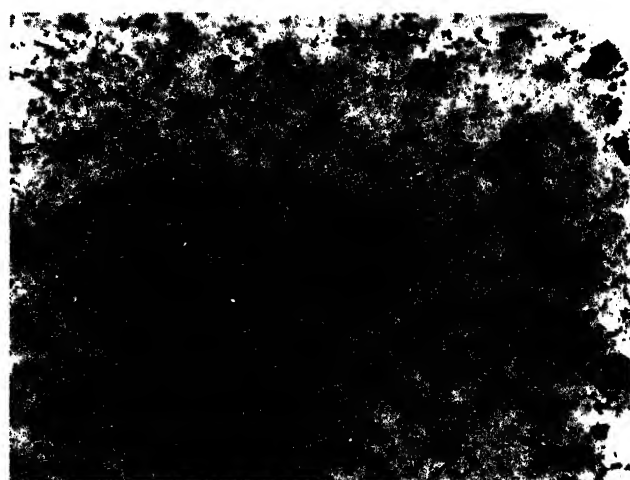
OAK RIDGE NATIONAL LABORATORY

DOE - \$200,000

10/18/74 - 10/18/76

Principal Investigator - B.R. Rodgers

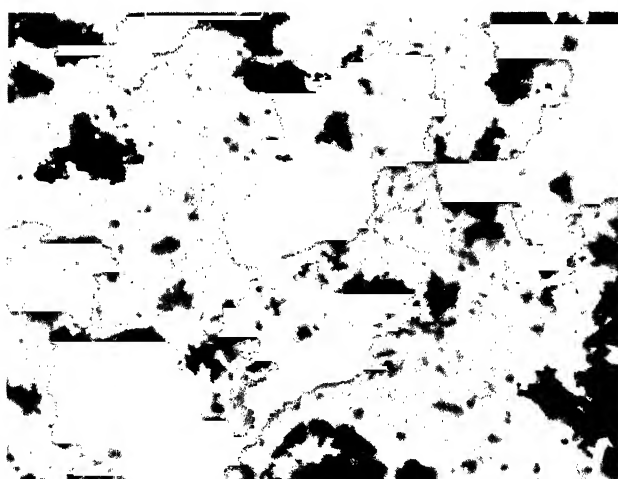
**OBJECTIVES** – This work involves performing research and development of improved methods for solid-liquid separations in coal liquefaction. Better understanding of the unique chemical and physical characteristics of coal liquids is essential to meeting this goal.



(a) NONAGGLOMERATION

SCALE - MICRONS

0 100 200



(b) AGGLOMERATION

SCALE - MICRONS

0 20 40

*Particles Present in Unfiltered Oil from SRC Processes (a) Can Be Agglomerated by Solvent Addition (b)*

**RECENT WORK AND ACCOMPLISHMENTS** – Laboratory experiments were conducted to characterize solids-containing streams produced by several coal conversion processes, including particle-size distributions, physical and chemical properties, petrographic analyses, neutron activation analyses, and photon emission spectroscopy data. Aging tests have determined the effects of sampling and storage under various conditions. Scouting experiments were used to explore various separation techniques that might apply to coal-derived liquids. The two most promising techniques (agglomeration followed by sedimentation, and filtration using carbonaceous filter media) were examined in a bench-scale unit. Initial feasibility and considerable economic incentives were demonstrated. A final report was prepared summarizing the results of this work and recommending further development.

**PLANS FOR THE COMING YEAR** – This contract was not extended by Fossil Energy; however, under sponsorship of the Division of Basic Energy Sciences, fundamental investigation will continue into the agglomeration of particulates in coal-derived liquids. The surface charge on particles will be measured in coal liquids, the relation of surface charge to particle agglomeration will be established, and the role of additives in promoting agglomeration through the modification of surface charge will be explored.

### CATALYST RESEARCH FOR COAL LIQUEFACTION

ENGELHARD MINERALS AND CHEMICALS CORPORATION

DOE - \$876,083; Engelhard - \$111,001

7/1/76 - 12/31/78

**OBJECTIVES** – This program is seeking to discover improved catalysts for the direct hydro-liquefaction of coal. Specific areas to be addressed by catalyst preparative work are optimization of pore-size distribution and improved hydrothermal stability of supports, and development of active constituents with enhanced activity and selectivity characteristics. The need for optimization of catalyst acidity also will be assessed. Supporting tasks include catalyst characterization, batch screening for initial activity and selectivity and evaluation of stability in a continuous mode of testing.

**RECENT WORK AND ACCOMPLISHMENTS** – Several aluminas have been prepared with surface areas varying from  $\sim 100$  to  $350 \text{ m}^2/\text{g}$ . These preparations vary from aluminas the total pore volume of which is in pores less than  $40 \text{ \AA}$  diameter to those with a major fraction in the range  $\geq 200 \text{ \AA}$ . Several were stabilized against hydrothermal sintering by introduction of various metallic cations. These supports were converted to catalysts (3 percent  $\text{CoO}$  - 15 percent  $\text{MoO}_3$ ) both with and without the addition of other metal oxides. A gas chromatographic method, based on programmed desorption of adsorbed bases (ammonia and pyridine) was developed and used to measure the surface acidity of these preparations. An autoclave system was designed, constructed, and put into routine use for catalyst evaluation (batch screening). Uncertainties in the measurement of reaction time and temperature normally encountered in batch operation have been reduced significantly via a novel design permitting coal injection after a slurry of catalyst in reaction solvent has reached operating temperature and by rapid cooling at termination. A test development program was carried out to define conditions required for evaluation of catalyst performance. A workup procedure that separates product into benzene-insoluble, pentane-insoluble (asphaltene), and pentane-soluble (oil) fractions was standardized. Screening conditions were selected based on a study of the effect of time, temperature, pressure, stirring speed, catalyst particle size, catalyst loading, and solvent/coal



ratio. Initial evaluation of commercial Co-Mo catalysts shows that they are not sufficiently selective in reducing the asphaltenic content of benzene-soluble products and are deficient in denitrogenation activity. Screening results indicate little beneficial effect of varying catalyst acidity. To overcome plugging problems normally encountered in downflow operations, an external-recycle, downflow fixed-bed reactor for longer term evaluation of promising candidates was designed and constructed.

**PLANS FOR THE COMING YEAR** – Goals of the preparative and screening work are optimization of support pore-size distribution and the identification of new compositions of active components leading to improved activity/selectivity behavior. Efforts will also be directed toward improving catalyst physical properties (density, abrasion resistance, etc.) in line with specific process requirements (e.g., H-Coal). Operability of the fixed-bed unit will be demonstrated, and the evaluation of promising candidates initiated.

## MECHANISMS OF HYDROGEN TRANSFER IN COAL HYDROGENATION

GULF RESEARCH & DEVELOPMENT COMPANY

DOE - \$776,895

6/29/76 - 6/28/79

**OBJECTIVES** – This project is seeking to understand the mechanisms of hydrogen transfer to coal in the basic liquefaction reaction environment used in processes being developed to convert coal to liquid. The project is divided into two 18-month phases, the first covering the study of model compounds and the second of applying these results to the study of coal and coal-derived liquids.

**RECENT WORK AND ACCOMPLISHMENTS** – Experimentation with model compounds is nearing completion. Runs were made with both tagged and untagged compounds as hydrogen donors and acceptors. It was shown that condensed aromatics were stable at liquefaction temperatures, while aromatic compounds linked with aliphatic or heteroatom groups typically present in coal cracked in the presence of a donor solvent. Detailed kinetic data were obtained for selected compounds and reaction mechanisms were suggested. The type of donor solvent strongly influenced the product distribution from the reactions. In addition, the solvent underwent rearrangement reactions that reduced hydrogen donor capacity. The use of tagged components has pointed out the importance of having good donor solvents in that most of the hydrogen being transferred to cracking components comes from the solvent and not from dissolved hydrogen.

**PLANS FOR THE COMING YEAR** – Emphasis will be on the study of hydrogen transfer to coal and coal-derived substances, specifically asphaltenes. Experimentation will be done with a wide range of reactor residence times, temperatures, and solvents of varying donor capacity. The use of deuterium,  $^{13}\text{C}$  and  $^{14}\text{C}$  tagged solvents will be included. The initial stages of liquefaction will be studied with deuterium, while the tagged solvents will be used to measure the extent of solvent/asphaltene or solvent/coal reaction resulting in the formation of adducts. The conclusions from the study of model compounds will be tested against those derived from the studies with asphaltenes and coal.

## DEUTERIUM TRACER METHOD FOR INVESTIGATING THE CHEMISTRY OF COAL HYDROGENATION

ROCKWELL INTERNATIONAL CORPORATION

DOE - \$152,199

6/26/76 - 6/28/77

**OBJECTIVES** – This research program involves developing and demonstrating a deuterium tracer method for investigating the mechanism of coal hydrogenation, and using the method to study important facets of coal hydrogenation. The ability to trace the incorporation of hydrogen into the coal structure is expected to aid in the evaluation of hydrogen donor solvents and in obtaining a better understanding of how such solvents and various hydrogenation catalysts work. This information may lead to conservation of costly hydrogen and reduction of coke formation. The ability to trace the incorporation of hydrogen should help to optimize coal hydrogenation process conditions and process design.

**RECENT WORK AND ACCOMPLISHMENTS** – A deuterium tracer method for investigating the chemistry of coal liquefaction has been developed and demonstrated. The method not only allows a study of deuterium incorporation into the coal structure from gas-phase deuterium ( $D_2$ ), but also an investigation of the deuterium uptake when tetralin- $d_{12}$  is used as a donor solvent. Hydrogenation experiments were conducted using powdered (-200 mesh) bituminous coal and deuterium to establish the degree and structural sites of deuterium incorporation into the coal structure under these baseline conditions before progressing to hydrogenation in the presence of a donor solvent and/or a catalyst. Parallel experiments, under identical experimental conditions, were also conducted with protium (the common isotope of hydrogen). In these experiments, the hydrogenated products were analyzed by proton nuclear magnetic resonance (NMR) spectroscopy. Since the deuterium incorporated into the coal does not absorb at the protium frequency, the difference between the two spectra gives a quantitative measure of the hydrogen that is taken up at different structural positions by the coal during hydrogenation. The deuterated products were also analyzed by deuterium NMR spectroscopy to obtain this information directly. The extent of deuterium incorporation is a function of time and temperature (44 and 19 percent D at 400°C and 380°C, respectively, over a reaction time of 1 hr/200 atm  $D_2$  pressure). Selectivity was observed in deuterium incorporation among the solvent-fractionated products. The deuterium concentration was lowest in the oil fraction. It was higher in the asphaltene fraction and highest in the benzene-methanol insoluble residue. No significant selectivity in deuterium incorporation was observed in any of the product fractions with respect to structural positions as determined by deuterium NMR spectroscopy. These deuterium distributions suggest that a considerable fraction of the soluble products are formed by thermal decomposition of the coal followed by internal hydrogen rearrangement (without reaction with  $D_2$ ). In preparation for hydrogenation experiments using tetralin- $d_{12}$  as the donor solvent, naphthalene- $d_8$  was prepared by the reaction of naphthalene with deuterio-phosphoric acid-boron trifluoride complex. Naphthalene- $d_8$  was converted to tetralin- $d_{12}$  by nickel-catalyzed reaction under deuterium pressure. The prepared tetralin- $d_{12}$  was then used as a donor solvent in a coal hydrogenation experiment. The experiment was carried out at 400°C and 200 atm pressure, using a 1-hr reaction time. No selectivity in deuterium pickup was observed with respect to the solvent-fractionated products; however, some structural selectivity with respect to a  $\alpha$ -carbon adjacent to an aromatic ring and the  $\alpha^2$ -carbon (or methylene bridge between two aromatic rings) was observed via deuterium NMR analysis.

**PLANS FOR THE COMING YEAR** – The study of the interaction of deuterium and coal in the absence of donor solvent or catalyst will be concluded and documented. The mechanism of hydrogen incorporation into coal in the presence of a hydrogen donor solvent will continue to be investigated using tetralin-d<sub>12</sub> and deuterium. The application of the deuterium tracer method to catalytic coal hydrogenation will also be investigated, both in the presence and absence of a donor solvent.

## EXPLORATORY RESEARCH ON CATALYSTS AND CATALYST SUPPORTS

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$250,000

1976 - Continuing

Principal Investigators - C.L. Kibby, A.J. Perrotta

**OBJECTIVES** – The goal is to develop new catalysts and catalyst supports suitable for synthetic fuels processing to produce various types of fuel. Considerable efforts have been directed toward developing new and better catalysts for petroleum refining by depositing active components on either alumina or silica. Only very recently has significant progress been made in catalysis in the area of catalyst support structure. To illustrate, zeolitic chemistry made a breakthrough in developing catalytic cracking and hydrocracking catalysts. A new breakthrough must come in the development of catalyst materials for the production of synthetic fuels from coal.

**RECENT WORK AND ACCOMPLISHMENTS** – A phase with composition  $\text{CoMo}_2\text{S}_4$  is formed in the Co, Mo, S system; this composition is close to the one used in successful hydrodesulfurization catalysts. PERC researchers are attempting to increase the yield and substitute Ni for Co. About 20 preparations in the Co, Mo, S ternary system, 15 in the Ni, Mo, S system, and 12 in the Ni, Co, Mo, S system have been made. The latter were at the composition  $(\text{Ni, Co})\text{Mo}_2\text{S}_4$ . These materials are being characterized. TGA reduction-oxidation measurements on nickel catalysts have been made so that metal dispersions can be calculated from volumetric  $\text{H}_2$  chemisorption data on the same catalysts. Volumetric  $\text{H}_2$  chemisorption data on a standard  $\text{Ni}/\text{Al}_2\text{O}_3$  catalyst reduced at 450°C give a nickel surface area of 4.2 m<sup>2</sup>/g. TGA measurements show a reduced nickel content of 64 mg/g; thus, the ratio of surface nickel atoms to total nickel atoms is 0.1, and the average crystallite size is 15 nm. Similar data are being obtained for Raney nickel samples after thermal sintering.

**PLANS FOR THE COMING YEAR** – This project has been combined with Coal Liquids Upgrading. The objectives for this portion of the combined project will remain the same. Work will center around catalysts and supports that are effective for hydrogenation and hydrocracking coal liquids in the presence of hydrogen sulfide and basic nitrogen compounds.

## NEW CATALYTIC PROCESSES FOR CHEMICALS

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$135,000

10/1/76 - 9/30/77

Principal Investigators - H.R. Appell, F.W. Steffgen

**OBJECTIVES** – The goal is to find new catalysts and reaction conditions for converting coal liquids or raw coal to large volume chemicals, such as aromatics, phenolics, and low molecular weight olefins. Also, the aim is to prepare and test new catalytic compositions that can be used to produce chemicals from coal and coal liquids.

**RECENT WORK AND ACCOMPLISHMENTS** — Several bituminous coals have been found to liquefy readily under pressure with hydrogen or synthesis gas at a temperature of 425° to 450°C and produce a low-viscosity product. Several percent of light oil can be distilled from this product, which contains much BTX and higher aromatics. Various catalysts can be added during the liquefaction step to increase the yield of light oil; for example, with an hvCb coal from the Iowa Demonstration Mine No. 1, ammonium chloride was found to increase the light oil yield to 7 percent. FIA analysis and MS-GLC analysis indicate that the light oil was about 70 percent aromatic. Thus, about 5 percent of the coal-derived oil was C<sub>6</sub>-C<sub>10</sub> aromatics. It is conceivable that a coal liquefaction process to produce fuel oil that feeds a reactive coal capable of yielding several percent of C<sub>6</sub>-C<sub>10</sub> aromatics would lend itself to the economic separation of important amounts of BTX and other chemical products. Experimental work on depolymerization of polystyrene using selected catalysts in a pulse flow microreactor was conducted to gain an understanding of how carbon-carbon bonds in large molecules break. This work could be related to large molecules in coal that must depolymerize to form chemicals of low molecular weight. Hydrogen at 50 psig was found to inhibit the reaction in the absence of catalysts (i.e., over glass wool). Sulfided CoMo catalyst, the only hydrogenation catalyst tested, produced 93 percent ethylbenzene and 7 percent styrene; however, when styrene was injected over sulfided CoMo, only 38 percent was hydrogenated to ethylbenzene. This fact indicates that intermediates in the depolymerization process are more readily hydrogenated than the monomer.

**PLANS FOR THE COMING YEAR** — The project was not funded for FY 1978.

## INFRARED SPECTROSCOPIC STUDIES OF ADSORBED SPECIES ON CATALYSTS

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$100,000

10/1/76 - 9/30/77

Principal Investigators - A.G. Sharkey, Jr., F.R. Brown

**OBJECTIVES** — This project is seeking to determine the nature of adsorbed reactants, intermediates, and products during reaction on coal conversion catalysts in an effort to deduce the mechanisms of catalyst deactivation.

**RECENT WORK AND ACCOMPLISHMENTS** — Emphasis has been placed on hydrodesulfurization and hydroliquefaction (supported molybdena) catalysts for the direct conversion of coal into environmentally acceptable liquid fuels. Primary effort has been directed toward the study of: commercial and in-house synthesized catalysts for their molybdenum-oxygen and -sulfur structural features, adsorbed compounds on the surface of such catalysts, and used catalysts removed from coal liquefaction reactors. The Raman spectra of over 75 molybdena catalysts have been recorded and interpreted. Fundamental questions concerning the nature of such catalysts have been addressed. The impact of surface area of the support, amount of molybdenum incorporated, effect of promoters, order of addition of active component and calcination temperature on the structural features of the catalysts have all been studied. Some of these catalysts have been sulfided and their structures determined. Infrared and Raman spectra have been used to monitor the nature of adsorbed pyridine and thiophene on the surface of selected molybdena catalysts. Correlations between the structure of the catalysts and the nature of the adsorbed heterocycles have been attempted, although more experimental data are necessary to validate the correlations. Raman spectra of used molybdena catalysts are dominated by intense scattering assigned to coke formed in

the pores of the catalysts. No bands assignable to molybdenum-oxygen structures of the catalyst proper were observed. Only trace amounts of molybdenum-sulfur species were noted, indicating that the formed coke was masking those structures from the Raman analysis and, presumably, from the organic sulfur species of the coal. It is postulated that this contributes to the observed deactivation of the catalysts.

**PLANS FOR THE COMING YEAR** – This project has been terminated.

## **SOLIDS SEPARATION BY SUPERCRITICAL GAS EXTRACTION**

**PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$100,000**

**1977 - Continuing**

**Principal Investigators - P.M. Yavorsky, S.E. Rogers**

**OBJECTIVES** – The objective of this project is to determine the feasibility of separating residual solid material from raw coal liquefaction products by using a solvent at pressures and temperatures above its critical point. Under these gas-phase conditions, the dissolving power of the solvent is greatly increased, and, in comparison with conventional vacuum distillation or pyrolysis, significantly greater recovery of ash-free coal-derived liquids can be attained.

**RECENT WORK AND ACCOMPLISHMENTS** – A bench-scale semicontinuous process unit was constructed and shakedown tests conducted using both raw liquid product and centrifuge residue from the Synthoil process as substrates and pure benzene and isopropyl alcohol as critical gas solvents. Preliminary results of these tests indicated that essentially all the liquids were recovered and that the remaining solid residue was a dry, friable powder.

**PLANS FOR THE COMING YEAR** – Emphasis will be placed on preparation and evaluation of solvents consisting of complex multicomponent mixtures derived from the product streams of various coal liquefaction processes. Also, a bench-scale unit will be designed and built to demonstrate operation of the process in a wholly continuous mode and to define the optimum process variables.

## **HOMOGENEOUS CATALYTIC REACTIONS**

**PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$75,000**

**1975 - Continuing**

**Principal Investigators - S.J. Metlin, F.W. Steffgen**

**OBJECTIVES** – This project has the goal of developing a practical process for the conversion of methanol to ethanol by homogeneous catalysis. The synthesis of other chemicals and low molecular weight fuels by similar catalytic systems using synthesis gas alone will also be investigated. The homologation reaction of methanol and synthesis gas produces ethanol, propanol, and low molecular weight hydrocarbons that can be subsequently converted to olefins (ethylene, propylene) using known technology or used directly as fuels and chemicals. This process will provide an alternate source of important chemicals, such as ethylene, now obtained almost totally from petroleum.

**RECENT WORK AND ACCOMPLISHMENTS** – The homologation reaction was discovered in this laboratory in 1949. The oil embargo of 1973 and the resulting chemical shortage led to renewed interest in the reaction, and this program was undertaken to achieve a practical route to make ethylene from coal. The original reaction used  $\text{CO}_2 (\text{CO})_8$  as the catalyst, with reaction pressures of 6000 psi at  $185^\circ\text{C}$ . The effects of various forms of platinum group metal promoters, both alone and in combination, have been investigated in PERC's laboratories during the past year. The most effective catalyst system discovered is a ternary mixture of  $\text{CO}_2 (\text{CO})_8$ , NaI, and  $\text{RuCl}_3$ . In batch experiments at pressures of 4000 psig and a residence time of 1 hour, methanol can be converted to ethanol in 50 percent yield at 70 percent selectivity. The effect of process variables, including including pressure, temperature, gas composition, catalyst concentration, and run duration have been investigated. Temperatures of  $220^\circ\text{C}$  or more destroy the catalyst while temperatures as low as  $140^\circ\text{C}$  shift the product distribution to methyl and ethyl acetates. Only slightly higher ethanol yields result from the use of 2:1 rather than 1:1 ( $\text{H}_2$ :CO) synthesis gas. Little effect on product distribution is caused by wide variation in catalyst concentration. Runs made in a rapid heating stirred autoclave at times from 0 to 6 hours show that short-duration experiments of 1 hour and less produce high ethanol yields. The reaction rate is second order with respect to the unconverted methanol, and according to the rate data, should proceed very well in a flow system at relatively short contact times.

**PLANS FOR THE COMING YEAR** – The investigation of new catalyst-promoter combinations will be continued in an effort to increase ethanol selectivity. Catalyst recovery and recycle and the use of solvents will be investigated for the preferred catalyst systems. The reaction mechanism will be studied further to improve the catalyst system and the overall reaction efficiency. A pressure flow unit will be used to study reactions over selected homogeneous as well as heterogeneous catalyst systems.

## STRUCTURAL CHANGES DURING COAL LIQUEFACTION

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$250,000

1975 - Continuing

Principal Investigators - A.G. Sharkey, Jr., H.L. Retcofsky

**OBJECTIVES** – This project involves application of advanced analytical techniques to support the development of coal conversion processes, and the development of instrumental techniques to provide needed analytical data to other projects at PERC. The project combines the long-range goal of elucidating the basic chemistry of coal liquefaction with necessary short-term goals, such as determining the causes of catalyst deactivation and the reasons for product instability during storage and transportation of the fuel.

**RECENT WORK AND ACCOMPLISHMENTS** – Knowledge of the changes in chemical structure that occur during coal liquefaction will assist in elucidating the basic chemistry of liquefaction processes and provide a scientific basis for selecting optimum conditions for plant operation. Data being obtained for coals and their liquefaction products include carbon aromaticities, characterization by chemical class, quantity and nature of free radicals, and heteroatom content. Spectral studies of asphaltenes showed that charge transfer interactions are relatively unimportant as binding forces between the acid and base components and that the aromaticity of asphaltenes is lower than that of the starting coal. The free spin concentration in the asphaltenes is approximately an order of

magnitude less than that in the original coal and is concentrated in the basic components. Investigations into the physical and chemical properties of centrifuged liquid product (CLP) from the 1/2-t/d Synthoil PDU are important in determining the factors affecting its stability during storage. Samples of CLP were aged under controlled conditions of temperature, atmosphere, light, and mechanical agitation while monitoring viscosity. The viscosity of all the samples increased with time; the samples having a higher initial viscosity increased more rapidly. Measurements of the rate of oxygen consumption, and data from solvent separation analysis of the aged product, indicate that most of the oxygen is incorporated into the asphaltene fraction. It was determined that oxygen-containing moieties, particularly hydroxyl groups, are most susceptible to oxidative degradation. Further characterization of the heteroatom species in coal liquids, which appear to influence catalyst deactivation as well as product stability, has shown that phenolic oxygen and basic nitrogen compounds predominate; cyclic ethers and pyrrolic nitrogen species are minor constituents. The nitrogen analogs of all the major aromatic ring systems associated with coal conversion products were found in the coal liquefaction product.

**PLANS FOR THE COMING YEAR** – The determination of heteroatom distribution in liquefaction products will continue with emphasis on relating changes in composition to changes in process variables. The role of free radicals in liquefaction mechanisms and in the aging of liquefaction products will be investigated. Used catalyst and inert packing material from sacrificed reactor sections will be analyzed to determine causes of catalyst deactivation.

## CHEMICAL STRUCTURE OF COAL AND COAL-DERIVED PRODUCTS

ARGONNE NATIONAL LABORATORY  
DOE - \$55,000  
10/1/76 - Continuing

**OBJECTIVES** – This project is using recently developed chemical and instrumental techniques to structurally characterize bituminous coal and its solvent refined product (SRC), and a lignite coal and its solvent refined product (SRL). From these studies, it is hoped to obtain a better understanding of the structural changes that occur during primary liquefaction.

**RECENT WORK AND ACCOMPLISHMENTS** – Samples studied in detail were: Illinois coal, Pittsburgh coal and its SRC product, Wyoming lignite, and North Dakota lignite and its SRL product. Each was characterized by elemental analysis; identification of constituents in fractions separated according to volatility and solvent solubilities (hexane, benzene-methanol, and pyridine); and identification of constituents resulting from a variety of oxidative degradations. Although the lignites were much less aromatic than the bituminous coals (Illinois and Pittsburgh), both solvent refined products were very similar with a greater degree of aromaticity than either feed coal. Thus, it appears that much greater degradation occurred during the solvent refining of lignite than of bituminous coal. The SRC process removed sulfur, but the SRL process introduced aromatic sulfur into the organic matrix of the product.

**PLANS FOR THE COMING YEAR** – These studies will be completed, and a detailed report comparing SRC, SRL, and petroleum residues will be prepared with emphasis on the significance for coal utilization.

## THERMODYNAMIC PROPERTIES FOR SELECTED KEY COMPOUNDS

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$100,000

1976 - Continuing

Principal Investigators - D.W. Scott, S.H. Lee-Bechtold, N.K. Smith

**OBJECTIVES** – This project is obtaining experimental values of enthalpy of combustion, heat capacity, and vapor pressure of selected key compounds important in the processing of liquids derived from coal. Emphasis is given to polynuclear aromatic hydrocarbons and their hydrogenation products and to similar compounds containing heteroatoms such as nitrogen. The thermodynamic information obtained may be useful in predicting the course of reaction in chemical processing.

**RECENT WORK AND ACCOMPLISHMENTS** – One of the first questions to be answered is that of the likelihood of further hydrogenation or cracking when a molecule such as 9, 10-dihydroanthracene is hydrogenated. The likely product of hydrogenation is 1, 2, 3, 4-tetrahydroanthracene. The product of cracking from the 9, 10-dehydro derivative would be tolyphenylmethane. Measurements of the heat of combustion and vapor pressure of 1, 2, 3, 4-tetrahydroanthracene were completed. Synthesis and purification of 9, 10-dihydroanthracene and tolyphenylmethane were completed at Oklahoma State University under the direction of Professor E.J. Eisenbraun. Calibration of a low-temperature adiabatic calorimeter is in progress. This calorimeter will be used for heat-capacity measurements on isoquinoline.

**PLANS FOR THE COMING YEAR** – Measurement of the thermodynamic properties of isoquinoline, 9, 10-dihydroanthracene, and tolyphenylmethane will continue.

## HEAT CAPACITIES AND HEATS OF COMBUSTION OF CHARS AND LIQUIDS

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$75,000

1975 - Continuing

Principal Investigators - S.H. Lee-Bechtold, W.D. Good

**OBJECTIVES** – Reliable thermal efficiency calculations for pilot or demonstration plants require information on the thermodynamic properties of the gaseous, liquid, and solid products. The objectives of this project are to provide heat capacity and enthalpy data on chars and liquids derived from coal and to relate these properties to the composition and physical properties of the parent coal, and to process variables in the reactor in which they arose.

**RECENT WORK AND ACCOMPLISHMENTS** – Measurements were made of heat capacity and heat of combustion of six chars of different origin: one was produced from Illinois No. 6 coal by the HYGAS process; a second was derived from western Kentucky coal by the COED process; two were derived from North Dakota lignite and two were derived from New Mexico bituminous coal at GFERC. Measurements of the heat of combustion were made at 298°K, and values of the heat capacity were obtained from 273° to 695°K. Measurements were made of the heat capacity and heat of combustion of four fractions of synthoil derived from West Virginia coal and three fractions of (COED process) syncrude derived from Utah coal. Values of the heat of combustion are referred to 298°K, and values of the heat capacity were obtained from 273° to 695°K (792°F) for the various fractions.



**PLANS FOR THE COMING YEAR** — Measurements of heat capacity and heat of combustion will be completed for liquid fractions derived from Hydrocarbon Research, Inc., syncrude and from a series of liquid fractions obtained from Conoco Coal Development Company. Enough experimental measurements will have been completed by the end of this year that attention can be given to correlation of thermodynamic properties with chemical composition.

#### **FUNDAMENTAL DATA NEEDS FOR COAL CONVERSION TECHNOLOGY**

**RECON SYSTEMS, INC.**  
DOE - \$97,608  
8/76 - 9/77

**OBJECTIVES** — This study is seeking specific process areas where new and/or more accurate thermodynamic data are needed for improved design and efficient operation of coal conversion processes. Specific recommendations for research to fulfill those needs will be made.

**RECENT WORK AND ACCOMPLISHMENTS** — The contractor has completed the study and submitted specific recommendations for research. A report has been presented orally and in written form. It has been generally accepted, but a few sections will be expanded and an Executive Summary provided.

**PLANS FOR THE COMING YEAR** — The final version of the written report should be completed early in the fiscal year.

#### **COAL CONVERSION PROCESSES—HYDROGEN SOLUBILITY**

**BARTLESVILLE ENERGY RESEARCH CENTER**  
DOE - \$75,000  
1977 - Continuing  
Principal Investigator - R.H. Harrison

**OBJECTIVES** — The solubility of hydrogen (and possibly synthesis gas) in selected coal liquefaction process solvents is being measured. These solubility data are needed to understand, develop, and commercialize coal conversion processes.

**RECENT WORK AND ACCOMPLISHMENTS** — The project proceeded from the initial analysis and research planning to the shakedown operation of the assembled apparatus. A research method was selected, equipment was ordered, the apparatus was assembled, and some shakedown measurements have been made on the solubility of helium in tetralin.

**PLANS FOR THE COMING YEAR** — The solubility of hydrogen in tetralin will be measured at several operating variables to test the apparatus and compare the results with literature values. The solubility of hydrogen will be measured in a simple, four-component synthetic solvent mixture and then in selected multicomponent coal-liquefaction-process solvents. Measurements will be made up to a temperature of 800°F and a pressure of 4000 psi.

## CHEMISTRY AND STRUCTURE OF COAL

OAK RIDGE NATIONAL LABORATORY

DOE - \$75,000

1976 - Continuing

Principal Investigator - V.F. Raaen

**OBJECTIVES** – This work involves the determination of the relative hydrogen-donor capabilities of various coals with respect to each other and with respect to hydrogen-donor compounds of the tetralin type.

**RECENT WORK AND ACCOMPLISHMENTS** – The organic compounds believed to be good hydrogen acceptors or aromatizing reagents are the quinones (e.g., chloranil); however, other classes of carbonyl compounds can also oxidize tetralin and other hydrogen donors. Contrary to Brower's conclusion, benzaldehyde does oxidize tetralin to naphthalene at 400°C in a Pyrex tube, and although decarbonylation appears to be the predominant reaction, some toluene is also formed. Tetralin-1-<sup>14</sup>C was used in the experiment and, during 17 hr of heating, underwent some cracking to methyl-<sup>14</sup>C-toluene. Benzophenone also proved to be a good hydrogen acceptor, undergoing only modest (1 to 4 percent hydrogenolysis to toluene during 24-hr heating at 400°C. Data were obtained under two sets of conditions: short-term heating (1- to 2-hr) at 400°C with hydrogen-donor in excess to determine the relative rates at which donors give up hydrogen, and long-term heating (24-hr) with benzophenone in excess to determine how much hydrogen can be obtained. Data from hydrogen-donor response for short-term heating are given in percentages as follows: The total percentage of hydrogen that can be removed from the coals studied is highest (up to 35 percent) for the bituminous coals, is less (22 to 24 percent) for the subbituminous coals, such as Wyodak, and becomes minimal (4 to 5 percent), as expected, for anthracite, which is more aromatic. The results resemble those found in the extensive investigation by Wender and co-workers of the catalytic dehydrogenation of a large number of coals. Although preheating coal may profoundly affect its solubility characteristics, preheating does not significantly affect its ability to give up hydrogen.

**PLANS FOR THE COMING YEAR** – The experiments just described will be expanded and refined to include other coals. Carbon-14 labeled reagents will be synthesized and allowed to react with various fractions from coal liquefaction processes to determine chemical and structural changes that take place.

## ELEMENTAL ANALYSIS OF COAL AND COAL ASH

AMES LABORATORY

DOE - \$30,000

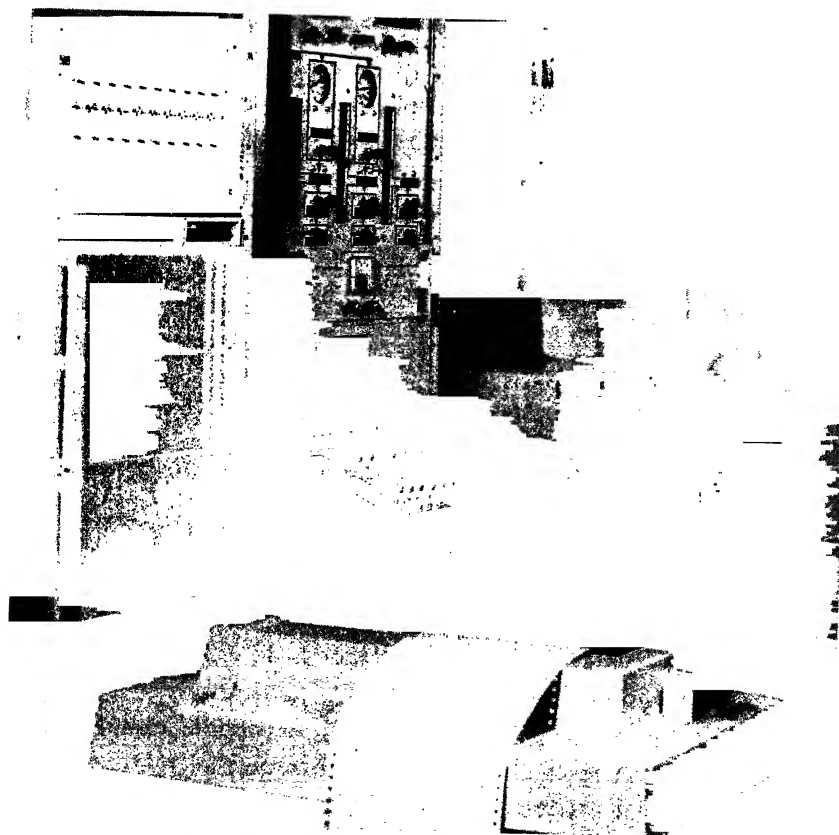
1/1/76 - Continuing

**OBJECTIVES** – This project is optimizing a new analytical spectroscopic method for the simultaneous determination of major, minor, and trace elements in coals and coal ashes without the excessive chemical preparation and separations commonly required. Increasing reliance on coal as a fuel or chemical stock places increased demands upon a reliable quantitative analysis of the inorganic concomitants of coal, for toxicological and environmental reasons as well as for catalytic or corrosive effects upon industrial processes. Not only is it important to know the composition of major elements, but concentration values for minor and trace elements must also be monitored since utilization prospects estimate thousands of tons of coal per year per industrial site. A simul-

taneous, multielement spectroscopic method of analysis has been developed here. The inductively coupled argon plasma (ICAP) has proven to be an excellent vaporization-atomization-excitation source for atomic emission spectroscopy (AES) and has been successfully employed with multi-element polychromators. Computer assisted data acquisition and reduction has eliminated much of the tedium of traditional spectroscopy. The facility can simultaneously determine concentrations of 20 elements, with a routine capability of 100 or more samples per day. Furthermore, a technician can be trained to operate the instrument in less than a week.

**RECENT WORK AND ACCOMPLISHMENTS** – A metaborate fusion followed by dissolution in dilute acid has proven to be a useful general method for obtaining solutions of all standards and samples that have been analyzed. The ICAP-AES method has been shown to provide reliable analytical data for a variety of standard samples for major elements A, Ca, Fe, Mg, Si, minor elements Ti and Cr, and trace elements Ba, Mn, and V. Good correlations have also been obtained with analyzed coal and ash samples received from Pennsylvania State University and the Illinois State Geological Survey. Linear calibration curves over three or more decades of concentration are regularly obtained, and service analyses for these elements are commonly performed.

**PLANS FOR THE COMING YEAR** – Alternative dissolution methods will be examined for the most useful means of obtaining more concentrated sample solutions. Additional minor elements and trace elements, which have shown analytical promise through probing experiments, will be studied further to define useful concentration ranges for analysis. Ultrasonic nebulization will be included in the procedure in a continuing program of optimizing powers of detection.



*Inductively Coupled Plasma Spectrograph*

## DEVELOPMENT OF NMR METHODS FOR ANALYSIS OF COAL

LAWRENCE BERKELEY LABORATORY

DOE - \$55,000

10/1/77 - Continuing

Principal Investigator - A. Pines

**OBJECTIVES** – Nuclear magnetic resonance (NMR) techniques are being developed to analyze directly the deuterium type in solid materials. Spectroscopic techniques are hardly applicable to whole coals and to solid residues in processing, so only small soluble fractions can be observed. For carbon, it has been shown that four major functional groups can be distinguished directly in the solid state. Similar developments for deuterium would allow the characterization of hydrogen types. Furthermore, the evolution of hydrogen during processing could be monitored to aid in understanding the detailed mechanisms involved in coal liquefaction.

**RECENT WORK AND ACCOMPLISHMENTS** – A deuterium probe with high-power RF and magic angle sample spinning has been constructed. The probe has a 4-kHz air-driven rotor and fiber optics to illuminate the sample for synchronization with spectrometer logic. The first deuterium chemical shift anisotropy has been measured by double-quantum NMR in the solid state. The experiment was done for benzene, and the measurement yielded  $\Delta\sigma = -6$  ppm, which is larger than theoretical estimates, thus making analysis of aromatic hydrogen for coal purposes more optimistic. Similar measurements were made for ferrocene and durene. The first high-resolution deuterium spectra in the solid have been obtained. The isotropic shifts of several different deuterium nuclei in model compounds were observed. The resolution between aromatic and aliphatic deuterium nuclei in perdeuterated dimethyl and diethyl terephthalate makes the possibility of deuterium analysis feasible, although it is anticipated that a higher magnetic field is needed.

**PLANS FOR THE COMING YEAR** – The limitation to resolution is the second-order quadrupole shift, which can be alleviated somewhat using higher fields. Accordingly, a high-field device is under design. At the current field, measurements of deuterium chemical shift anisotropies will continue by double-quantum NMR to provide a basis for analytical purposes as has been done for carbon by double resonance. When the high-field unit is operational, work will begin on model compounds for coal and for real coal processing samples from other DOE labs.

## CHEMICAL ANALYSIS AND EVALUATION OF COAL SAMPLES

U.S. GEOLOGICAL SURVEY

DOE - \$336,000; U.S.G.S. - \$2,200,000

7/1/76 - 9/30/77

**OBJECTIVES** – This program is providing part of the necessary chemical and physical data on coal resources needed for planning, designing, siting, and constructing coal conversion and combustion plants slated to come on stream in the late 1970's and early 1980's. The impact of such data is primarily in four domains: matching feedstock resources with processes, assessing environmental impact, appraising technological options, and evaluating byproduct recovery potential.

**RECENT WORK AND ACCOMPLISHMENTS** – Approximately 1400 channel coal samples from coal beds throughout the United States were collected and analyzed under this program. Of these samples, 850 were Eastern coals and 550 Western coals. They are derived from thick and thin beds;

high and low sulfur coals; coal with full range of ash contents; and ranks ranging from lignite to Massachusetts meta-anthracites. The samples are being entered into an accessible computer data system.

**PLANS FOR THE COMING YEAR** — By the end of FY 1977, some 3200 coal samples will have been analyzed. The original chemical and physical properties of raw coal either directly or indirectly affect: handling and pretreatment of feedstock prior to energy conversion; devising techniques for the recovery and/or disposal of potentially harmful trace elements such as As, Sb, Se, and Hg from the gas, liquid, and solid effluent produced during energy conversion; and technological problems such as corrosion, erosion, and clogging of plant plumbing, and poisoning of catalysts. The geochemical and physical data resulting from this program will provide a starting point for assessing these items and detailed material balance studies in all conversion and combustion processes.

## **MECHANISM OF COAL LIQUEFACTION**

### **PITTSBURGH ENERGY RESEARCH CENTER**

DOE - \$225,000

1974 - Continuing

Principal Investigators - B.C. Bockrath, S. Friedman, F.W. Steffgen

**OBJECTIVES** — This work is seeking to determine the mechanism of coal liquefaction, coal hydrodesulfurization, and CoMo catalyst deactivation, and to provide a basis for further improvements in coal liquefaction technology. The mechanism in the Synthoil process may be separated into reactions that proceed with a CoMo catalyst and those not requiring this catalyst. Conversion of asphaltenes to oil and removal of their heteroatoms can be accelerated by a number of catalysts. In particular, the viscosity of the product oil depends largely upon its asphaltene content, and conversion of asphaltene to oil is a critical parameter in maintaining product uniformity.

**RECENT WORK AND ACCOMPLISHMENTS** — A pulse flow microreactor was used to study spent catalyst taken from the Synthoil 1/2-t/d continuous process unit. This used catalyst was less active than fresh sulfided catalyst for conversion of low-molecular-weight model compounds; however, conversions of model compounds over carefully regenerated catalyst are nearly the same as with fresh catalyst. Thus, coke formation appears to be the major cause of catalyst deactivation, as measured by catalytic reactions of low-molecular-weight model compounds. The thermal and catalytic conversions of asphaltenes to oils were studied in a batch autoclave. To obtain more detailed information about the transformation of asphaltenes, gel permeation chromatography (GPC) was used for obtaining a small number of distinctive fractions. After analysis, both the total conversions of asphaltenes to oil and the relative conversions of the nonpolar and polar fractions were compared. Total conversions are increased by higher reaction temperatures, the presence of CoMo catalyst, or longer reaction times, although reaction rates fall off with times, the polar compounds constitute a smaller fraction of the asphaltenes. Thus, the residual asphaltenes are more refractory, less polar, and more aromatic than those in liquid products before extensive hydrotreatment. GPC was also used to compare asphaltenes and their fractions on the basis of molecular size; for example, asphaltenes derived from autoclave liquefaction of an Iowa coal were found to be, on the average, smaller in size than those produced in pilot plant experiments from either Western Kentucky or West Virginia coal. In related work concerning the possible effects of molecular size on catalytic processing, it was found that asphaltenes derived from the Kentucky bituminous coal were not excluded from the microporous surface areas of a fresh sample of a CoMo hydrodesulfurization

catalyst. Thus, physical exclusion on the basis of molecular size should not be a factor in catalytic processing, at least not with fresh catalyst.

Viscosity is one of the more important characteristics of coal liquefaction products. Factors that influence viscosity were studied by preparing reconstituted liquids from the pentane soluble oils and either toluene insolubles, asphaltenes, or the subfractions of the asphaltenes. On a weight basis, toluene insolubles influence viscosity more than asphaltenes. Molecular aggregation, either among the acidic and basic subfractions of asphaltenes or between toluene insolubles and asphaltenes, increases the viscosity of the liquids.

**PLANS FOR THE COMING YEAR** – The role of native coal mineral matter as a catalyst in the primary liquefaction reactions will be studied with the use of both coal and model compounds. The chemistry of the pyridine soluble-benzene insoluble fraction, and also of the asphaltenes, will be studied by making molecular modifications by methods such as reductive alkylation or acetylation. Studies to determine the controlling factors of product oil viscosity will be continued. Advanced separation and analytical techniques to support these studies will also be developed.

#### EXTRACTIVE SEPARATION OF ASH FROM COAL LIQUIDS

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$200,000

1976 - Continuing

Principal Investigators - C. Ortuglio, J.G. Walters

**OBJECTIVES** – The goal is to determine if extractive separation of ash and unreacted coal from coal liquefaction product oil is a viable method of recovering product oil from the primary separation residue. Up to a third of the product oil may be trapped in the primary separation residue and must be recovered to improve the economics of the coal liquefaction process.

**RECENT WORK AND ACCOMPLISHMENTS** – Various solvents have been investigated with respect to rate of solvation, temperature, degree of agitation, and quantity of solvent required to determine the effect of these variables on the settling time of the ash and unreacted coal. Basic data from these experiments are being used to design a bench-scale separation system to obtain engineering data. Immiscible solvent pairs were tested to determine if a satisfactory separation of ash could be obtained, and several solvent pairs produced promising results. Bench-scale equipment is being constructed to provide better evaluation of these solvent pairs.

**PLANS FOR THE COMING YEAR** – A bench-scale countercurrent liquid-liquid solvent extraction unit is being fabricated to evaluate the efficiency of the most promising immiscible solvent pairs. Preliminary basic investigation of froth flotation as a separation method will be initiated, in addition to a continuing survey of the efficiency of solvents and flocculents.

## DEASHING OF SOLVENT REFINED COAL

LAWRENCE BERKELEY LABORATORY

DOE - \$30,000

1976 - Continuing

Principal Investigators - R. Steninger II, C. Radke, D. Hanson

**OBJECTIVES** – This program is seeking to determine the most promising route of development for ash removal in the SRC process. The work has concentrated on processes to alter the surface properties of the particles and thereby effect agglomeration. Solving this problem by agglomeration techniques should result in an economically feasible method to replace filtering techniques such as the diatomaceous earth filters now being used.

**RECENT WORK AND ACCOMPLISHMENTS** – Pyridine has been determined to be a fluid in which agglomerated ash particles can be resuspended and have the same characteristics as the SRC unfiltered oil. The colorless pyridine allows standard turbidity monitoring, resulting in a rapid, meaningful screening test for the coagulation process. Three surface altering processes are being studied: *charge neutralization coagulation*—initial candidates that have been screened include aerosol OT, Cu and Zn aleate, Ca dodecylsulfate, and Ca solicylate; *bridging flocculation*—initial studies of this method in nonaqueous media have included looking at oil soluble dispersants and latex emulsifiers such as polystyrene, actylphenoxylethanols, acreloid dispersants, and naphthalene sulfonic acids; and *water scavenging*—methods are being screened to transfer the particles to a bulk aqueous phase from which they can be separated by ordinary means such as filtration, centrifugation, or evaporation. The approach is based on altering the surface properties of the particles through a surface active agent to make them hydrophilic.

**PLANS FOR THE COMING YEAR** – Promising results from the current investigation of agglomeration in pyridine will be verified by high-temperature settling in SRC unfiltered oil. Next, if high-temperature filtration results are promising, consideration will be given to specification of the optimum sequence of separation steps involving settling, filtration, and possible centrifuging.

## CROSS-FLOW FILTRATION FOR SOLIDS REMOVAL FROM COAL SYNCRUDES

WALDEN DIVISION OF ABCOR, INC.

DOE - \$176,983

2/76 - 3/78

**OBJECTIVES** – This program initially investigated the application of cross-flow filters to the removal of solids from SRC filter feed; however, it became apparent that cross-flow filtration might be more suitably applied to polishing overflow from solvent deashing units. After completing the experimental program with SRC filter feed, initial screening tests were conducted to investigate the cross-flow filtration of solvent deashing unit overflow.

**RECENT WORK AND ACCOMPLISHMENTS** – Cross-flow filtration tests were conducted on samples of SRC filter feed obtained from Pittsburg & Midway Coal Mining Company, Tacoma, Washington and solvent deashing overflow obtained from Conoco Coal Development Company, Library, Pennsylvania. Preliminary analyses were made of the SRC filter feed to characterize the viscosity-temperature relationship, the distillation curve, the partial pressure-temperature relationship and the particle size distribution. No attempts were made to characterize the solvent deashing

overflow sample. The preferred operating conditions, based on test results at different temperatures, linear velocities through the filter tube (velocity), and filtration pressures were: temperature—maximum permissible (consistent with temperature limitations of equipment and feed material); velocity—20 ft/sec; and filtration pressure—35 psi. During a 216-hr batch concentration/life test in which SRC filter feed was processed at these conditions with a  $5\mu$  element, the flux declined from 1.2 gal/ft-hr initially to a very low level (0.12 gal/ft<sup>2</sup> hr) by the end of the test. Attempts to increase the flux by backflushing and solvent cleaning decreased in effectiveness as the test proceeded. The maximum volumetric concentration ratio obtained during the test (1.57) was limited by an apparent valve failure. The performance of the filter deteriorated with time, resulting in a time-averaged ash content in the filtrate of 0.113 wt percent, compared to 0.05 wt percent for earlier tests. At the conditions investigated, none of the flux regeneration techniques (in-situ backflushing, solvent forwardflushing/backflushing, and acid soaking) proved to be effective for restoring the flux to its original level. Several techniques were evaluated for increasing the flux and its stability with operating time. The most effective was the use of a microgranular prefill material (0.075 $\mu$  carbon black particles) to prevent the SRC solids from penetrating and plugging the porous elements. After 1-hr operation, the flux was approximately twice as great and much more stable than a similar element without prefill. Tests conducted to determine the feasibility of using cross-flow filtration to upgrade the quality of solvent deashing overflow (0.25 wt percent ash to <0.05 wt percent ash) were promising. Filtrate ash contents of 0.05 wt percent were obtained for all the elements tested, and flux levels were more than an order of magnitude greater than those obtained with SRC filter feed. (Integral average flux of 6.7 gal/ft-hr during 7 hours of operation compared with a value of 0.6 gal/ft-hr filtering SRC filter feed under the same operating conditions.) Based on the flux levels obtained during this program, cross-flow filtration does not appear to be an economically viable process for solids removal from SRC filter feeds; however, cross-flow filtration does appear to show potential for upgrading the quality of solvent deashing overflow.

**PLANS FOR THE COMING YEAR** — The final report for this program has been prepared and submitted, concluding the technical effort under the present contract.

## ANALYTICAL RESEARCH

### PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$150,000

1977 - Continuing

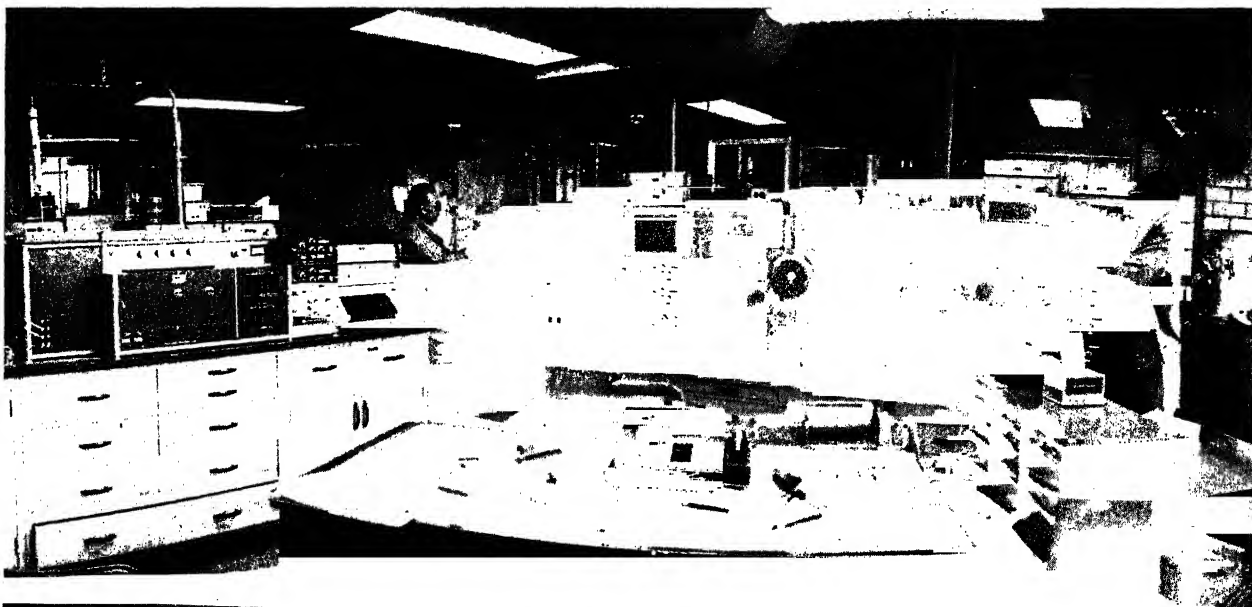
Principal Investigators - H. Shultz, M. Mima

**OBJECTIVES** — A standard method for the determination of asphaltenes in coal-derived liquid and solid fuels is being designed and tested and the fate of environmentally important trace elements in coal liquefaction determined. No standard method exists for the determination of asphaltenes in the products of coal conversion. Furthermore, there is no known relationship between asphaltene determinations obtained by analytical methods currently in use. Thus, comparison of products from various coal conversion processes is not rigorous. This work compares five different solvent separation methods used for determining asphaltenes in coal-derived products. Based on the results obtained in testing the five methods, a standard method incorporating the best features of the tested methods will be proposed.

**RECENT WORK AND ACCOMPLISHMENTS** — The first material chosen for the asphaltene tests was a centrifuged liquid product from the 1/2-t/d unit located at PERC. The second material chosen



was a solvent refined coal. The samples were divided into five representative portions and stored under appropriate conditions. Testing of the first three methods (i.e., twenty replicate analyses) was completed on the liquid product and work on the fourth method initiated on the same material. The procedures for each method have been followed faithfully. Data collected include weight percent asphaltenes, insolubles, and oils. Standard deviations are being calculated for each method, and statistical comparisons will be made between methods. Data collected on testing the first three methods indicate statistically significant differences in the results, which confirms the need for a standard method. A workshop attended by over 50 representatives of industry, university, and Government laboratories was arranged by PERC to discuss the status of asphaltene determinations. After developing appropriate analytical procedures for the determining six environmentally important trace elements, the 1/2-t/d Synthoil PDU was sampled and the samples analyzed. The distribution of trace Cd, Pb, Cr, Cu, Ni, and Mn in the PDU was determined and material balances performed. The major finding was that the centrifuge residue is the primary sink for the trace elements studied.



*Flameless Atomic Absorption Spectrometer for Determination of Trace Metals in Coal Conversion Process Streams*

**PLANS FOR THE COMING YEAR** — Replication of the fourth and fifth asphaltene methods, using the centrifuged liquid product, will be completed. All five methods will then be tested in the same manner on the sample of SRC and statistical comparisons made. After reviewing the results, a standard method will be formulated for further testing. The trace element investigations will continue with determinations on streams from the Synthane gasifier.

#### **PROCESS RESEARCH DIGEST**

**OAK RIDGE NATIONAL LABORATORY**

DOE - \$65,000

7/1/76 - Continuing

**OBJECTIVES** — This work provides reviews of coal conversion processes and technology of current interest to individuals engaged or interested in fossil energy research.

**RECENT WORK AND ACCOMPLISHMENTS** – First-issue topics and related materials for the digest have been selected. Procedures, publication assistance, organization, writing, and review of the articles are essentially complete. Articles to be included in the first issue are: "Coal Gasification with Chemically Incorporated Catalysts," "Flash Hydropyrolysis of Coal," "Zinc Chloride Hydrocracking of Coal and Coal Extracts," and "Conversion of Methanol to High-Octane Gasoline." Articles for the second issue have been selected. Material collection and writing are in progress.

**PLANS FOR THE COMING YEAR** – The first issue of the digest will be published in November 1977. Second-issue publication is scheduled for mid-1978.

## HOMOGENEOUS CATALYTIC UPGRADING

ARGONNE NATIONAL LABORATORY

DOE - \$45,000

1976 - Continuing

**OBJECTIVES** – This project will develop the information used to assess the technical and economic feasibility of a new concept for upgrading the products of existing coal liquefaction processes. At some point in all processes, the coal extract is high melting and/or extremely viscous, high in mineral matter and nitrogen content, and of inadequate volatility or heating value for use as a distillate or as a boiler fuel. These problems can be overcome by further, separate process steps to remove mineral matter and to accomplish secondary hydrogenation in catalytic reactors; however, the methods are expensive and technically difficult. A new concept, in which both steps are accomplished together by means of a recyclable liquid catalyst system and low-temperature synthesis gas, offers the potential of more economical coal-liquefaction processes.

**RECENT WORK AND ACCOMPLISHMENTS** – Bench-scale (1/2-gal) equipment was built and tested. With SRC-1 filter feed as typical test material, six runs were made to determine operability of the catalytic hydrogenation. It was established that hydrogen uptake could be readily followed by the installed pressure-measuring devices; that partial recycle of product would be required to achieve the desired liquid-liquid separation; that density, viscosity, distillability, and heating value were all improved; and that mineral matter separation was occurring. In addition, sensitivity studies showed that liquid catalyst losses of up to 1 percent/turnaround were economically tolerable.

**PLANS FOR THE COMING YEAR** – The equipment will be modified to include a three-phase (mineral matter rich, liquid catalyst, product oil) separation device. Process variable studies will be performed for optimizing the equipment with respect to residence time and product properties, and at least one other feedstock will be examined. Catalyst recovery will be quantitatively determined. The results will be evaluated in terms of a decision to halt, to continue bench-scale studies, or to plan for larger scale continuous operation.

## REFINING AND UPGRADING OF DIFFERENT SYNCRUDES TO TRANSPORTATION FUELS

CHEVRON RESEARCH COMPANY

DOE - \$1,588,598

6/30/76 - 2/27/79

**OBJECTIVES** – This program is determining the feasibility and estimating the economics of hydroprocessing four syncrudes to distillate fuels using available technology. The first two feedstocks are

a Colorado shale oil from an aboveground retort and SRC produced in the Pittsburg & Midway Coal Mining Company pilot plant at Tacoma, Washington. The remaining feeds will be a catalytic hydroliquefaction liquid to be produced by the H-Coal process and a fourth synthetic liquid to be determined by mutual agreement between DOE and Chevron. The results will provide the basis for an overall refining plan, plant cost estimates, utility and hydrogen requirements, and the like. Tests will be conducted to develop supporting information for estimating commercial plant performances using existing technology.

**RECENT WORK AND ACCOMPLISHMENTS** – In shale oil processing, the first feedstock to be evaluated is Paraho crude shale oil, produced in the indirectly heated mode. Shale oil is unusually high in nitrogen content compared to petroleum distillates; therefore, work has emphasized nitrogen removal from the whole shale oil by hydrotreating. Several catalysts were evaluated for this purpose using a fixed bed, and ICR 106 was selected as the catalyst of choice. With it, nitrogen can be reduced from 2.2 percent to quantities as low as 1 part per million in a single catalytic stage; however, the economic optimum appears to be in the range of 500 to 1000 ppm nitrogen in the whole liquid product. Hydrotreated shale oil has an advantage as a refinery feed since it contains essentially no residuum and very little sulfur. The feasibility of hydrotreating whole shale oil was demonstrated in several pilot plant tests, including a 3700-hr test that was shut down when the feed supply was exhausted. The results show that commercial length runs can be expected. The 650°F+ fraction of hydrotreated shale oil is an excellent feed for a fluid catalytic cracker. Pilot catalytic cracking studies show that gasolines and cycle oils thus derived are similar to those obtained from the cracking of hydrotreated petroleum gas oils. Studies are also in progress in which hydrotreated shale oil is being converted to jet fuel and gasoline in an extinction recycle hydrocracker. An alternate processing scheme also pilot tested is one in which the shale oil is coked, and the resulting coker distillate is hydrotreated to produce diesel and naphtha. Based on correlations, the naphthas from the shale oil hydrotreater can readily be upgraded to high-octane gasolines by catalytic reforming. The middle distillate fractions may require some additional hydrotreating to produce salable diesel or jet fuel. Pilot plant studies have been made to verify performance of a middle distillate hydrotreater. Overall, study results are encouraging in that shale oil appears to be an attractive feed for processing in existing refineries that have appropriate hydrotreating facilities.

On the other hand, SRC is difficult to process. Because of its high melting point, it must be dissolved in an appropriate solvent before hydrotreating in existing fixed-bed pilot plant equipment. The first solvent for SRC in these tests was a 50/50 blend in creosote oil. In the first test, ICR 106 catalyst showed essentially no fouling in 330 hr. About half of the 850°F+ SRC was converted to 850°F- distillate, and the nitrogen content was reduced from 1.5 percent in the SRC/creosote oil blend to 0.2 to 0.3 percent in the product; however, after 330-hr on-stream, plugging occurred in the catalyst bed. In a second run, 1100-hr operation was achieved without plugging; however, the catalyst deactivation rate was relatively high. The 350° to 850°F product from the SRC/creosote oil hydroprocessing runs was tested. Plugging problems were again encountered using this as the solvent for SRC. Downstream processing studies with distillate product from the hydroprocessed SRC/creosote oil blend are in progress.

**PLANS FOR THE COMING YEAR** – Evaluation of the results of the pilot plant tests for processing of shale oil and SRC will be completed. Work on the remaining two feedstocks will be started when they are supplied by the DOE.

**RECENT WORK AND ACCOMPLISHMENTS** — An instrumented 1-in.-I.D. by 12-ft-long downflow entrained tubular reactor has been designed and constructed. The unit has been operated at up to 2 lb/hr flow of coal, 3 lb/hr of hydrogen, 850°C, reaction temperature, and 4000 psi pressure. An on-line gas chromatograph analyzes gaseous and liquid products at various points along the length of the reactor. Liquid products are condensed, measured, and analyzed. The conversion of lignite to products as a function of temperature, pressure, residence time, feed gas composition, and gas-to-coal-feed ratio has been determined. Material balance closures for the major elements in coal including C, H, O, S, and N are obtained. Approximately 100 runs have been made during FY 1977 using North Dakota lignite. The major products are liquid BTX and heavier hydrocarbons ( $\geq C_9$ ), gaseous methane and ethane ( $< C_5$ ), and carbon oxides (CO and CO<sub>2</sub>), as well as char. The liquid yields reach a maximum in the range of 750° to 800°C at pressures of 2000 to 3500 psig with conversion to 10 percent BTX, 10 percent oils, 36 percent HC gas, and 6 percent CO for a total of 62 percent carbon conversion. Above 800°C at pressures to 4000 psi, the liquid products decrease and the gaseous products increase with total conversion reaching 82 percent. A preliminary parametric economic evaluation of a full-scale flash hydropyrolysis plant has been performed. Above 60 percent conversion, which is the hydrogen balance point, the profitability of the plant remains about the same.

**PLANS FOR THE COMING YEAR** — Improvements in the operation of the experimental unit will be made including coal feeding and liquid product analysis. The product yields as a function of residence time will be determined over the pressure and temperature range of 500 to 4000 psi and 500° to 850°C. The yield with recycled char and ash, subbituminous and bituminous coal, and varying gas composition also will be determined. Exploratory runs with catalyzed coal will be made.

## SELECTED CONVERSION OF SYNTHESIS GAS FROM COAL TO HIGH-OCTANE GASOLINE

MOBIL RESEARCH AND DEVELOPMENT CORPORATION  
DOE - \$1,396,847; Mobil - \$349,219  
7/1/76 - 6/30/78

**OBJECTIVES** — This study involves the development of a catalytic process for converting synthesis gas of the type produced in coal gasification to a high-octane gasoline based on a Mobil proprietary catalyst. It is exploratory in nature and both single- and two-stage cascade processes are being considered. Specific objectives include the definition of commercially feasible process conditions, the effects of a variety of feed compositions, catalyst aging rates, the effects of a variety of feed compositions, catalyst aging rates, the effects of various catalyst regeneration procedures, the analyses of gas and liquid products, and the measurement of octane number and distillation range of the liquid product.

**RECENT WORK AND ACCOMPLISHMENTS** — Originally, both adiabatic fixed-bed and fluid-bed processes were considered for development; however, reactor plugging caused by carbon deposition and/or heat removal problems resulted in elimination of the fixed-bed process from consideration. Two catalyst compositions are being used for the process development: one converts 80 percent of synthesis gas of H<sub>2</sub>/CO composition with a selectivity of about 60 percent to C<sub>5</sub>-400°F hydrocarbons having a clear research octane number of 93; the other converts 90 percent of a 2H<sub>2</sub>/CO gas mixture with a selectivity of 75 percent to a C<sub>5</sub>-400°F product of 84 octane number. The current effort is devoted to establishing aging cycle times and regeneration procedures for these catalysts. Completed theoretical studies include: thermodynamic equilibrium for carbon formation, relation-

ship of single pass to total conversion, water-gas shift activity of specific catalysts, heat-loss calculations for the bench-scale fluid unit, and a heat and mass transfer study.

**PLANS FOR THE COMING YEAR** – Work will continue on multiple regenerations of promising catalysts. Both oxidative and hydrogenative regeneration procedures will be considered. Following identification of a viable catalyst composition, optimization of process variables such as feed composition, temperature, pressure, and space velocity will be completed.

## **SUPPORT COAL GASIFICATION (CATALYTIC GASIFICATION)**

**PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$150,000**

**10/1/76 - Continuing**

**Principal Investigator - W.P. Haynes**

**OBJECTIVES** – The program is designed to provide support for DOE contracts on catalytic gasification and to develop a third generation catalytic coal gasification process. Catalytic gasification represents a third generation process which promises significantly higher reaction rates resulting in smaller gasifiers or greater throughput. It is desirable to test independently different feedstocks, such as Battelle treated coal, in fairly large equipment to verify that pretreatment is accomplished and higher gasification rates result. It is also desirable to generate basic catalytic hydrogasification data to support DOE's catalytic gasification program as well as develop alternative processes.

**RECENT WORK AND ACCOMPLISHMENTS** – Bench-scale equipment to develop processing data for catalytic impregnation into coal and to obtain reactivity data of the catalytically impregnated coals have been designed, assembled, and operated. The impregnation process has been designed to be compatible with a slurry feed system. Chemicals that can reduce the agglomerating propensity of bituminous coals have been determined. Relative reactivities have been measured for several catalytically impregnated coals. Tests to verify the catalytic process undergoing development at Battelle Memorial Institute have been run. Computer programs to ascertain material balances on the reactivity tests have also been developed. Battelle treated coal made from Illinois No. 6 coal has been run in the Synthane PDU. This test indicated that the Battelle treated coal had a higher reactivity than mildly oxidized coal and was non-caking. Steady state has not been achieved; therefore, the tests are not considered conclusive.

**PLANS FOR THE COMING YEAR** – Equipment will be reconstructed to obtain larger samples of impregnated coals with greater flexibility in processing. The bench-scale hydrogasifier will be outfitted with a computer-controlled gas chromatograph and flow control equipment for greater reliability and ease of operation. Process development of impregnation will continue, emphasizing more complete impregnation of the catalytic materials into the coal structure. A new reactor will be designed and built to operate at higher temperatures and pressures. The Synthane PDU will be used to test large batches of catalytically impregnated coals including Battelle treated coal.

# CATALYTIC CONVERSION OF COAL ENERGY TO HYDROGEN

TRW SYSTEMS  
DOE - \$363,492  
6/1/76 - 12/31/77

**OBJECTIVES** – This program primarily seeks to develop a preliminary assessment of the potential of using selected alkali salt-based catalyst systems for promoting coal char gasification reactions involving steam with and without a carbon oxide acceptor. A key part of the effort involves the identification and development of catalysts which show a high activity and selectivity in promoting steam-char-acceptor reactions and are capable of maintaining their high activity for extended periods of performance. Catalytic coal gasification offers the possible advantages of significantly improving the technical and economic viability of producing useful synthetic fuels from coal. Through the development and use of effective stabilized catalysts, several relatively expensive unit operations such as water-gas shift reactors, methanators, oxygen plants, and special high efficiency acid gas scrubbers that are commonly a part of a conversion process, may be eliminated or significantly reduced in scale.

**RECENT WORK AND ACCOMPLISHMENTS** – The effectiveness and functional lifetimes (recyclability) of several alkali catalyst systems were evaluated for the coal char-steam-carbon dioxide acceptor reaction system using laboratory-scale fixed and fluid-bed reactors. In addition the effects of elevated pressure on char gasification rate and hydrogen yield for the catalyzed char-acceptor-steam reaction were preliminarily determined. The effects of catalyst application technique, concentration, and acceptor regeneration treatment on catalyst performance properties were also assessed. Evaluation of alkali catalyst performance properties for the char-steam (non-acceptor) reaction and the char-oxygen-steam reaction was also initiated. High char gasification rates were shown to be obtainable with sodium and potassium carbonate based catalyst systems for reaction temperatures in the 650° - 700°C range. This reaction temperature range is approximately 150 to 200° lower than the temperatures required to effect rapid reaction rates for uncatalyzed char gasification. In batch fluid bed reactor experiments, a 95 percent pure hydrogen product could be obtained from the catalyzed char-steam-acceptor reactions using modest reaction pressures of 3 to 6 atm. Further, it was shown that these high reaction rates and product yields are achieved through simple admixing of 5 to 10 percent dry catalyst powders with char or char-acceptor mixtures. In recycle experiments where reaction residues from steam gasification reactions were remixed with fresh char, alkali catalyst systems could be used to catalytically gasify between 12 and 35 times their weight of char before losing their catalyst activity. Recyclability performance appeared to depend on catalyst and char type, a 950° - 1000°C acceptor regeneration treatment, and the presence of lime and stabilizing additives such as fluorspar and phosphate salts. Volatilization and conversion of alkali catalysts to less active forms were shown to be possible mechanisms in the loss of catalyst activity with recycle. A conceptual process design based on alkali catalyzed char-acceptor-steam gasification reactions has been developed and preliminary economic comparisons with other coal conversion processes were completed. The results of these studies indicate that the catalytic process for producing a high purity hydrogen product could be competitive on a product cost per energy unit basis with other advanced coal conversion processes producing different fuel products.

**PLANS FOR THE COMING YEAR** – Experimental work will include elevated pressure fluidized-bed tests aimed at assessing the effects of residence time, pressure, steam and oxygen partial

pressure, and catalyst-to-acceptor ratio on gasification rates and product gas distributions. Catalyst performance with a variety of char types will be studied using a fixed bed reactor and an effort made to identify catalyst and reaction product intermediates involved in the catalyzed gasification reactions. The preliminary conceptual process design will be updated to reflect new experimental information developed on catalyst performance properties.

## IMPROVED METHANATION SYSTEM FOR COAL GASIFICATION

MIDLAND-ROSS TECHNICAL CENTER  
DOE - \$249,547; Midland-Ross - \$132,068  
4/1/76 - 3/31/78

**OBJECTIVES** – This program is designed to develop an improved methanation boiler, specifically for use in coal gasification plants. The program is directed at (1) elimination of the need to recycle product gas for temperature control, (2) making approximately 90 percent of the reaction heat available for producing high pressure (greater than 1000 psia) steam, (3) reducing operating costs by eliminating recompression and providing high steam quality, and (4) reducing capital cost by combining the methanation with the already required heat exchangers. The design concept evolved by approaching the methanator as a major source of energy and specifying a combined heat transfer and catalytic reaction system to optimize the availability of high temperature heat. The concept utilizes a patented catalytically coated insert placed inside heat exchanger tubes. The reaction heat is transferred to boiling water surrounding the tubes by combined convection and radiation. The insert is designed to maximize the heat transfer temperature differentials. The specific objective of this program is to test the insert methanator concept in a single tube section and to obtain design and operating data to allow proper insert selection, more accurate economic evaluation, and scaling to multitube units. This includes a computer simulation of the heat and mass transfer in the reacting system, and a literature survey. The experimental results will include reaction rates, temperature profiles, heat transfer rates, pressure drops, and stability measurements for several different inserts. The primary variables will be insert shape, catalyst activity, mass flow rate, and reactant concentration. The computer simulation will be calibrated from the measured data, and then will be used to extend the results. The program is funded through initial proof of concept testing.

**RECENT WORK AND ACCOMPLISHMENTS** – At the end of FY 1977, the test facility was operating, and preliminary testing of the first catalytic insert was completed. The computer simulation was used to compare the theoretical data and experimental results. The test facility is capable of evaluating an insert that fits into a 2 inch ID section with a 5 ft active length. The test section is cooled by Dowtherm operating at a nominal 550°F. The auxiliary system supplies up to 350 scf/m of preheated reaction gas at 500°F, and 1000 psia, containing up to 18 percent CO. The insert temperature profile and extent of reaction are used to evaluate the insert reactivity. The test section is too short to provide complete reaction, but the entire length of a full sized unit can be simulated by changing the inlet conditions. The auxiliary equipment includes compressors, heat exchangers, Dowtherm system, and a gas generator to produce CO and H<sub>2</sub> from natural gas. The present insert configuration consists of Raney nickel sprayed onto a metallic substrate. The substrate has a central



**PLANS FOR THE COMING YEAR** – The present test program will conclude when two insert designs have been tested in the facility. The test results will be analyzed using the computer simulation for the final report.

## **METHANATION CATALYST DEVELOPMENT**

**PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$100,000**

**1971 - Continuing**

**Principal Investigators - R.A. Diffenbach, H.R. Appell**

**OBJECTIVES** – Factors which affect stability, selectivity, and activity of methanation catalysts will be determined, and improved catalysts for the Synthane process will be developed. Raney nickel catalysts have shown excellent activity in tube-wall reactors for converting synthesis gas to methane. There appear to be several possible deactivation mechanisms of this catalyst. For an orderly development of improved catalysts, it is necessary to understand the major means of catalyst deactivation.

**RECENT WORK AND ACCOMPLISHMENTS** – An investigation of catalyst deactivation at typical methanation conditions showed that the loss of activity of Raney nickel catalyst proceeded rapidly in its initial stages of use followed by a slower rate of deactivation. This is interpreted as a combination of two deactivation mechanisms. The initial rapid loss of activity is believed to be a result of carbon deposition, whereas the slower rate of deactivation appears to be due to sintering.

**PLANS FOR THE COMING YEAR** – The investigation of Raney nickel catalyst will be redirected to developing new catalysts for synthesis of gasoline range liquids and chemical feedstocks.

## **TREATMENT OF COAL GASIFICATION WASTEWATER**

**PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$70,000**

**8/77 - Continuing**

**Principal Investigator - W.P. Haynes**

**OBJECTIVES** – Methods will be developed for treating coal conversion wastewaters (specifically Synthane gasification waters) that are capable of meeting legally established or proposed standards.

**RECENT WORK AND ACCOMPLISHMENTS** – The design of an integrated wastewater treatment system consisting of modules of various treatment steps is continuing. The system is being designed so that the modules or treatment steps can be switched to other positions in the treatment train or removed entirely. Design capacity of the system is about 1 gal/hr. Procurement of instrumentation and analytical equipment required for the integral unit has begun, and some of the equipment has been received.

**PLANS FOR THE COMING YEAR** – Design and construction of an integrated treatment unit will be completed. Operation of the unit with Synthane water from PDU at PERC will begin; later, treatment of wastewaters from other coal conversion pilot plants will be evaluated.



## GAS PURIFICATION IN COAL GASIFICATION PROCESSES

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$75,000

1977 - Continuing

Principal Investigator - L. Lorenzi, Jr.

**OBJECTIVES** – This project is designed to evaluate gas purification systems which are potentially applicable to coal gasification processes. Primary consideration is sulfur removal from the coal-derived gas to allow the satisfactory end-use application of the gas. Economic comparisons must be developed to identify systems bearing the greatest potential for minimizing the cost of the gas purification section of the overall coal gasification plant. The need for research to insure that purification technology is applicable to various alternative coal gasification system characteristics must be evaluated.

**RECENT WORK AND ACCOMPLISHMENTS** – Review of the literature on gas treatment systems and their performance capabilities has been completed. Gas purification process vendors, licensors, and design/constructors have been contacted to obtain process performance characteristics and preliminary economic estimates based upon specified gas purification requirements. Operators of existing pilot-scale facilities have been contacted for specific information regarding the technology employed for gas purification. The production and handling of (non-H<sub>2</sub>S) trace sulfur compounds are substantial factors in the design and operational performance of the gas purification system. These non-H<sub>2</sub>S sulfur species also become critical for control and removal due to potential environmental performance standards being developed for high-Btu coal gasification systems. The information gathered from literature studies and direct contacts is being assembled into a report. Conclusions and data evaluations which result from this assessment are being utilized to develop the specifications for detailed economic comparisons of alternative gas purification system options.

**PLANS FOR THE COMING YEAR** – Experimental support will be acquired for the adequate characterization of sulfur species in the purification train of coal gasification processes. This will include obtaining data on conversion of non-H<sub>2</sub>S sulfur species to H<sub>2</sub>S as a result of catalytic beds, purification operations, etc. A report presenting all findings of the literature studies and organizational contacts will be completed. Economic studies of alternative gas purification systems potentially applicable to coal gasification will be conducted. This information will be utilized to evaluate the potential for cost savings in the gas purification train of the overall coal gasification plant.

## ADVANCED GASIFICATION CONCEPTS

MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$125,000

10/76 - Continuing

Principal Investigator - L.A. Bissett

**TVES** – This study on coal gasification concepts is being made as an engineering assessment to (1) determine the technical feasibility and economic impact of novel processes in coal gasification, (2) identify and help initiate pertinent areas of research, and (3) increase the overall understanding of existing gasification processes with mathematical modeling. Such a study will also assist in the overall planning of gasification projects by a review of the state-of-the-art that will help identify high cost or problem areas associated with the technology.

**RECENT WORK AND ACCOMPLISHMENTS** – Review and summary of MERC entrainment gasification research during the 1950s and a general review dealing with the historical aspects and developments in entrainment gasification have been completed. A simplified mathematical model for entrainment gasification and thermodynamic and steady state differential models of fixed-bed gasification have been developed. Literature searches for fast fluidization, catalytic gasification, and flash pyrolysis with initiation of assessments have been completed, and mathematical modeling of previous MERC flash carbonization data has been initiated.

**PLANS FOR THE COMING YEAR** – The major assessment work on the various gasification concepts, entrainment, fast fluidization, catalytic and flash pyrolysis, will be completed by the end of the first quarter of FY 1978. Preparation of research proposals will follow along with the decision on the research area for experimentation. Flash carbonization and fixed-bed gasification modeling work will be completed as independent milestones.

### PARAMETRIC CHAR STUDIES

MORGANTOWN ENERGY RESEARCH CENTER  
DOE - \$175,000  
10/76 - Continuing

**OBJECTIVES** – This project will measure the physical and chemical characteristics and reactivities of chars produced from coal conversion processes, and to identify the best end-uses for these products.

**RECENT WORK AND ACCOMPLISHMENTS** – A thermogravimetric analyzer (TGA), having a sample capacity of 6 grams and capable of being operated at 1050°C and 1500 psig was purchased from Battelle Columbus Laboratories. A laboratory site was chosen, and a special high-pressure cell was designed and constructed for housing the TGA. A Varian model EM-600 mass spectrometer to measure TGA off-gas composition was purchased and installed. After installation and shakedown of the TGA, a series of experiments reacting carbon dioxide with Hydrane HY-128 char was begun. During the last quarter of FY 1977 the initial series of experiments using carbon dioxide as reactant gas was completed and a similar series using hydrogen was begun. The Analytical Division developed an improved method of determining surface properties of chars. A kinetic model for data analysis was obtained from a survey of the recent literature.

**PLANS FOR THE COMING YEAR** – The effects of sample basket mesh size, sample pelletization, and sample particle size on char reactivity will be studied. Hydrane, Synthane, and BIGAS char reactivities in hydrogen and steam will be determined and correlated to published models.

### RAPID HYDROGENATION FOR COAL CONVERSION TO SYNTHETIC MOTOR FUELS

INSTITUTE OF GAS TECHNOLOGY  
DOE - \$1,500,000  
4/1/76 - 6/1/80

**OBJECTIVES** – This program is designed to develop a noncatalytic process for the hydropyrolysis of lignites and coals in a short-residence-time entrained-flow reactor at high temperature and pressure for the production of fuel gases, high-octane gasoline blending stock, and fuel oil. The process under investigation is characterized by a relatively small volume reactor that is capable of processing

large volumes of feed materials at high space velocities. Thus, for a given processing capacity, the equipment needed for such an entrained-flow process would be of a smaller scale than the equipment needed for a comparable process using a fluidized bed or liquid-solid slurry, should be less costly, and should require less lead time for construction. When coal or lignite is heated quickly in a reducing atmosphere, gases, liquids, and tars are produced in a complex system of chemical and physical processes leaving a hydrogen-deficient char residue. In experimental work, the fraction of feed converted to gases and liquids has been found to exceed the "volatile matter" content of the lignite. The liquids are low in viscosity and are easily filtered to remove particulate matter. In the equipment train, the unreacted solids are disentrained from the reactor effluent at temperatures high enough to prevent recondensation of liquid and tars on the char, which is collected as a dry powder. The liquids are then condensed and separated from the methane, ethane, and carbon oxides and carrier hydrogen. The methane, ethane, and carbon monoxide would be recovered as intermediate products while the hydrogen would be recycled to the process. Unconverted char would be gasified to generate hydrogen for the process.

**RECENT WORK AND ACCOMPLISHMENTS** — Construction of the bench-scale unit has been completed; to date, approximately 40 runs have been made using a variety of operating conditions. At an operating pressure of 2000 psig, a coil outlet temperature of 1450° to 1500°F and a residence time of 2 to 3 seconds, approximately 50 percent of the feed carbon can be converted, with 13 to 14 percent of the carbon reporting as hydrocarbon liquids, 15 percent reporting as methane, 8 to 10 percent as ethane, and 8 percent as carbon oxides. There is evidence that some of the carbon oxides react with hydrogen to produce methane, but this effect is small. The choice of operating conditions has a strong influence on the distribution of compounds in the hydrocarbon liquids. When processing with low-thermal-severity conditions, substantial quantities of toluene and phenol will be found in the hydrocarbon liquids in addition to benzene, naphthalene, and other aromatic compounds. Under more severe thermal treatment, the yields of toluene, phenol, ethylbenzene, and xylene are reduced, with an increase in benzene yield. It is believed that this redistribution takes place through dealkylation and dehydroxylation. A limited amount of work has shown that char can be recycled to produce additional hydrocarbon liquids and gases.

**PLANS FOR THE COMING YEAR** — Experimental work in the bench-scale unit and PDU design will be continued. Further study will be made in the bench-scale unit of the effects of residence time, severity of thermal treatment, and operation with a simulated recycle carrier gas composed of hydrogen, carbon monoxide, and carbon dioxide. Work with steam and synthesis gas as carriers will also be done. Processing economics have been found to be sensitive to the choice of method used to separate product hydrocarbon gases from the hydrogen carrier; paper studies of established technologies such as oil scrubbing or cryogenic distillations, and newer technologies such as membrane separations will be made to explore methods for efficient separation and recovery of methane, ethane, and carbon oxides from the stripped carrier gas.

## **LOW-BTU GASES AS INDUSTRIAL PROCESS FUELS**

**INSTITUTE OF GAS TECHNOLOGY**  
DOE - \$833,530  
10/1/76 - 11/30/78

**OBJECTIVES** — The use of low- and medium-Btu gas is a promising way for industry to meet its need for an environmentally acceptable fuel to supplement or replace natural gas and oil.

Information is needed, however, to determine the extent of the problems of utilizing these fuels on existing equipment. This program is designed to develop the combustion data necessary for the Department of Energy (DOE) and industry to evaluate the feasibility of converting existing process heating equipment to low- and medium-Btu gases. Eight different types of industrial burners will be tested using three different low- and medium-Btu gases. The performance of each burner with these gases will be compared to its performance with natural gas.

**RECENT WORK AND ACCOMPLISHMENTS** – During the first part of the program, the IGT pilot-scale furnace was modified to allow the insertion of cooling tubes to provide an adjustable furnace load. The cooling tubes were fabricated, and the water inlet manifolds and return system were installed. Surface Combustion Division of Midland-Ross Corp. constructed a special gas-generating system for producing the necessary low- and medium-Btu gases. The gas-generator was installed and piped into the IGT furnace system. Gases simulating the composition of Koppers-Totzek oxygen (KTO), Wellman-Galusha air (WGA), and Winkler air (WA) fuel gases will be produced and tested. A Bloom baffle burner was installed as the first type of burner to be tested. The load was adjusted to simulate the preheat zone of a five-zone steel slab reheat furnace, and baseline data were gathered with a natural gas input of  $5.25 \times 10^6$  Btu/hour. KTO and WGA were fired on the same fuel nozzle with no instability problems; however, WA could only reach 60 percent of the natural gas heat input before becoming unstable (blowing off). Increasing the air preheat level from 650° to 800°F allowed WA to be fired at the same rate as the natural gas baseline. The combustion trials with natural gas, KTO, WGA, and WA included measurement of the thermal efficiency, heat absorption profile, flame shape, flame temperatures, resonance noise level, radiant heat flux, and flow direction profiles. The furnace efficiencies were 26 percent for KTO, 23 percent for WGA, and 22 percent for WA compared to 35 percent for natural gas. Peak flame temperatures were 1650°C for KTO, 1351°C for WGA, 1396°C for WA, and 1672°C for natural gas. All of the substitute fuels had longer flame lengths than the natural gas flame. Because the WA heat absorption profile and thermal efficiency were distinctly different from the natural gas measurements and due to the flame stability problem, combustion trials were begun with a larger fuel nozzle and a modified baffle.

**PLANS FOR THE COMING YEAR** – Testing of the seven remaining burners will be carried out. The burners will include a rotary kiln, nozzle mix, high forward momentum, flat flame, high excess air, premix tunnel, and boiler burner. At the completion of the project a manual will be prepared to inform the users of industrial burner equipment of the results of this study. This will aid them in determining if retrofitting is possible and what burner modification or furnace operation changes would be necessary.

## INSTRUMENTATION FOR ON-LINE ANALYSIS OF LOW-BTU GAS FROM COAL

AMES LABORATORY  
DOE - \$75,000  
3/1/77 - Continuing

**OBJECTIVES** – This program is being conducted to design and build instrumentation for determining sodium and potassium in the cleaned low-Btu gas derived from coal. Although the present work deals only with the clean gas stream, the ultimate objective is the analysis of the hot, uncleaned gas immediately downstream from the producer. Turbine failure is a problem when

low-Btu gas derived from coal is used as a fuel. This problem is related to the sodium and potassium content in the gas stream, and measurements upstream and downstream from the scrubbers are necessary to evaluate the effectiveness of the clean-up procedures. Instrumentation developed in this project should be adaptable to analysis of other fuel gas streams.

**RECENT WORK AND ACCOMPLISHMENTS** – A two-channel computer controlled flame emission spectrometer system has been built, and preliminary tests have been conducted using the gas stream from the Morgantown Energy Research Center (MERC) producer. The results of these tests indicate that there is little or no vapor phase sodium and that most of the sodium resides in small particulates which have a maximum effective diameter of a few microns. Detection limits are estimated to be in the fraction part-per-billion range provided contamination from the atmosphere can be eliminated. MERC is presently building a special clean room to house the instrument.

**PLANS FOR THE COMING YEAR** – Measurements of the sodium and potassium content of the MERC cleaned producer gas stream will be made after the clean room has been completed. Work will be started on a sequential multielement analyzer which will sample and analyze the gas immediately downstream from the MERC producer before cleanup. This instrument will automatically produce sequential analyses for up to 60 elements selected by the operator.

#### MEDIUM- AND HIGH-TEMPERATURE GAS CLEAN-UP OF PARTICULATES

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

DOE - \$150,300

9/27/77 - 9/26/79

**OBJECTIVES** – The applicability of the electrofluidized bed (EFB) to the collection of particulate products of coal combustion is being investigated. Bench-scale experiments are to span the range of 400°F to 1800°F covering conventional utility, fluidized-bed combustion systems, gasifier, and combined cycle system operating regimes. Particulate injection will allow injection of combustion products of a variety of coals or even combustible material if desired. From this program, suitable models for predicting the behavior of high-temperature EFBs and workable designs will be evolved.

**RECENT WORK AND ACCOMPLISHMENTS** – During the first 3 months of this 2-year program the design of the test rig has been completed and construction has begun. The test rig is being fabricated in stainless steel sections of 9-in.-by-9-in. cross-section with external thermal insulation. Natural gas burners will supply hot combustion gases which, along with injected particulate, will pass through the EFB.

**PLANS FOR THE COMING YEAR** – During the coming year the test rig will become operational. Charging techniques for the particulate will be developed and materials for the EFB construction will be selected and tested. By the end of the year many of the operational characteristics of the high-temperature EFB should be fairly well outlined.

## HAZARDOUS ELEMENTS IN SUPPLEMENTARY COAL FUELS

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$150,000

1975 - Continuing

Principal Investigators - A.G. Sharkey, Jr., R.G. Lett, C.E. Schmidt

**OBJECTIVES** – This research is being undertaken to support coal conversion processes by obtaining data on possible hazardous elements and compounds in process streams. The project will provide data that will assist in the design and implementation of purification systems for producing environmentally acceptable fuels from coal. The rapid development of coal liquefaction and gasification processes warrants a thorough evaluation of the potential hazards of coal conversion products with respect to end use and worker safety. Since many of the process development and scale up units will be self-contained, purification of major streams such as process wastewater is of considerable importance.

**RECENT WORK AND ACCOMPLISHMENTS** – All major process streams from the 1/2-t/d Synthoil PDU were surveyed for possible hazardous elements and compounds. Each stream was analyzed for 60 to 70 trace and minor elements and screened for the possible presence of several hundred potentially hazardous compounds. Coal liquefaction product oils from the start, middle, and end of two runs in the 1/2-t/d Synthoil PDU were also screened. In both cases the number of possible hazardous compounds increased as the run progressed. Process streams including the feed coal, water, tar, char, and filter ash from two Synthane PDU gasifier runs were surveyed for trace and minor elements. The results indicate that most of the trace elements were retained in the gasifier char; however, the gas filter ash contained higher relative concentrations of several volatile elements such as Cd, Pb, and As than the corresponding coal or char. In the case of the condensate water, potentially hazardous trace metals were generally at the sub-ppm level. Analytical data were also obtained on influent and effluent samples from two Synthane condensate wastewater treatment processes to determine specificities and removal efficiencies for trace organic contaminants.

**PLANS FOR THE COMING YEAR** – As process variables are changed, coal conversion process streams will be examined for toxic or otherwise hazardous compounds.

## TECHNICAL SUPPORT SERVICES

RADIAN CORPORATION

DOE - \$966,900

6/76 - 6/79

**OBJECTIVES** – This program will provide technical support to Division of Coal Conversion personnel responsible for technical and environmental aspects of coal conversion demonstration and pilot plant projects. This support is in the form of assuring that (1) all basic factors that should be considered in the early stages of a program are identified for planning via guideline documents, (2) interim results on programs are technically acceptable and indicate logical progress toward ultimate program goals, (3) any item identified as being critical to the viability of a coal conversion project is investigated, and (4) preparation or review of pertinent information can be made in a timely manner.

**RECENT WORK AND ACCOMPLISHMENTS** – Site suitability and water availability reports were provided for two coal conversion demonstration projects—ICGG Pipeline Gas and Conoco Pipeline Gas. In addition, a water availability assessment was prepared for the Coalcon project. Environmental review was provided for the Coalcon process until the termination of that project. A report was prepared identifying potential socioeconomic effects resulting from the possible shutdown of a demonstration plant, and a document was prepared identifying factors to be considered in evaluating the suitability of any site proposed for a demo project. Technical reviews were provided on the interim results of conceptual designs for atmospheric fluidized-bed steam generating plants.

**PLANS FOR THE COMING YEAR** - Guidelines describing the operational environmental characterization methodology for coal conversion plants will be completed and incorporated into an environmental monitoring handbook for coal conversion demonstration plants being prepared by Oak Ridge National Laboratory. The safety and health items associated with development of coal conversion facilities will be reviewed, and appropriate monitoring methods identified. Also environmental control technology for coal conversion demonstration plants will be identified as well as selection criteria for choosing specific control technologies. Information from the latter two efforts will assist the Division of Coal Conversion in the assessment of the developing technologies. The review and evaluation of conceptual designs for AFBC steam-generating plants will also be completed this year. As coal conversion plant projects move from the early planning to the design stage, interim review support will be provided for these projects. Water availability studies will also be performed for upcoming demonstration plant projects. Continued technical and environmental support to the Division of Coal Conversion will be provided on a quick-response basis.

## **TABLES OF THERMODYNAMIC DATA FOR SELECTED CHEMICAL SPECIES**

**THE DOW CHEMICAL COMPANY**

**DOE - \$260,000**

**9/1/76 - 8/31/79**

**OBJECTIVES** – Through a critical evaluation of pertinent literature, tables of thermodynamic data for compounds associated with coal conversion and coal combustion processes are being developed. This program will provide a set of thermodynamic data that will facilitate the interpretation, correlation, and extrapolation of experimental process results.

**RECENT WORK AND ACCOMPLISHMENTS** – Data coverage will include species occurring in fossil fuel processes such as coal gasification and combustion; slag vaporization; boiler and gas turbine corrosion; steam turbine, gas turbine, and magnetohydrodynamic power generation; sulfur control; and catalyst deactivation in methanation and direct liquefaction. Species covered include the rare gases and their unipositive ions, nickel sulfides, sulfuric acid (and its hydrates), alkaline earth sulfides, and nickel chloride.

**PLANS FOR THE COMING YEAR** – Pertinent literature will be reviewed and existing data evaluated for compounds of interest. Data will be published quarterly as part of the Joint Army-Navy-Air Force (JANAF) Thermochemical Tables.

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## ***DIRECT UTILIZATION***

### **CHARACTERIZATION OF THE CHEMICAL COMMUNUTION OF COAL**

**SYRACUSE RESEARCH CORPORATION**

**DOE - \$173,982**

**10/1/76 - 12/31/77**

**OBJECTIVES** — This program is determining the effect of physical and chemical properties of coal on the rate and nature of fracture induced by ammonia (gaseous and liquid) under various reaction conditions. It is also determining the influence of chemical treatment on the properties of the treated coal.

**RECENT WORK AND ACCOMPLISHMENTS** — Coal samples of various ranks—lignite to anthracite—were fragmented in anhydrous liquid ammonia, hydrous liquid ammonia, and gaseous ammonia under varying conditions of temperature, pressure, and exposure time. It is shown that coal of any rank can be fragmented, and the rate and degree can be very effectively controlled by changing the process variables. Also, the effect of the moisture content of coal on the fragmentation rate is rank dependent and varies with the process conditions. Chemically fragmented coal is shown to provide a good feedstock for above-ground gasification reactors. Chemical treatment reduces the swelling index of coal and converts a coking coal to a noncoking one. Tests have indicated that the hardgrove grindability of chemically treated coal is lower than that when the coal is not treated. Thus, costs incurred in reducing chemically treated coal to a size necessary for power generation will be considerably lower than for untreated coal. Comparative washability studies on mechanically crushed and chemically comminuted coal samples have shown the superior ability of the latter technique in liberating the pyritic sulfur and ash. The chemically comminuted coal has to be reduced to a much coarser size consist as compared to the mechanical crushed product to attain the same quality of the clean coal. It is shown that for any level of sulfur concentration (in the coal), substantially higher recoveries are attainable by chemically comminuting the coal. The combined effect of higher recoveries, less fines, and a cheaper separation flow sheet resulting from the coarser size consist of the product will more than make up for the increased costs of chemical treatment.

**PLANS FOR THE COMING YEAR** — The project ends this year.

### **EXCHANGE OF ASH AND SULFUR MONITORS**

**MORGANTOWN ENERGY RESEARCH CENTER**

**DOE - \$1,000**

**11/75 - 9/78**

**Principal Investigator - R. Stewart**

**OBJECTIVES** — A nuclear-based monitor for sulfur, developed by MERC, was sent to England for testing for possible application to their coals and their preparation plant conditions. In exchange, England's National Coal Board (NCB) has provided one of their Phase 3A nuclear-based ash monitors for testing with U.S. coals under preparation plant conditions. The testing includes evaluation for accuracy and serviceability for the application. In addition to preparation plants, such meters could have applicability with any large coal user since price and utilization control are based on quality of coal feed. The nuclear-based monitor concept has potential for other phases of coal assay that are being investigated.



**RECENT WORK AND ACCOMPLISHMENTS** – Negotiations for installation of NCB's Phase 3A ash meter at a nearby preparation plant were unproductive; therefore, it was installed in MERC's Recycle System, but several electrical malfunctions delayed initiating the tests. The problem has been corrected based on modifications to a similar Phase 3A unit installed at a Pittston Coal Company plant in Virginia. Reasonably stable operation has been attained and calibration started. The reason for the extensive calibration requirement for U.S. coal is in the different applications in the two countries. The British use the ash meter to help blend dry high-ash coal; here, its use would be primarily with cleaned, typically wet, low-ash coal. The ash meter has been found accurate to 1 to 2 percent ash in coal. The problem encountered in the U.S. installation is to maintain gravity-flow of wet material by the meter.

**PLANS FOR THE COMING YEAR** – Testing of the British ash meter will continue and the MERC sulfur meter will be evaluated in England for another year. MERC is monitoring other Phase 3A monitors installed in commercial plants. Part of the ash meter tests will compare these results with the MERC element meter. Other plans include calibration and comparison of a nuclear Btu meter system, composed of ash, sulfur, and moisture meters, with conventional analyses and Btu measurements on test coals.

#### **DEVELOPMENT OF A NUCLEAR ASSAY INSTRUMENT FOR COAL**

**MORGANTOWN ENERGY RESEARCH CENTER**

**DOE - \$190,000**

**9/76 - Continuing**

**Principal Investigator - R. Stewart**

**OBJECTIVES** – This project is developing a nuclear technique for direct determination of the composition of coal minerals as a basis for continuous monitoring of the heat value (Btu) of coal. Involved in this effort is the measurement of high-energy gamma rays, use of fast analog-digital electronics, and on-line measurement of individual elements in coal. This determination provides fast information toward evaluation of coal quality required for coal preparation and efficient utilization. The Btu determinations are useful for quality control purposes in coal-cleaning plants and in increasing thermal efficiencies of power plants.

**RECENT WORK AND ACCOMPLISHMENTS** – The on-line measurement of combustible content of coal is conceptually attractive and has now been shown to be technically feasible by MERC. The major and minor elements in coal—S, Fe, Al, Si, Na, K, Ca, Ti, Cl, and N—have been monitored separately as additives to coal. Prompt gamma ray spectra of each element, determined by a difference method, have been assimilated in a computer evaluation program to eliminate mutual interferences. The results were used to estimate the technical feasibility of a Btu meter by calculations based on measurements of moisture, sulfur, and a computer sum of major ash elements. The resulting Btu value, calculated from the Paar equation, is accurate to within 62 to 210 Btu (0.4 to 1.3 percent) depending on the rank and impurity level of the coal. These error limits are determined from the limits of detection for each measured component. A continuous, on-line Btu meter is technically feasible. Reasonable accuracy can be obtained from separate monitoring of moisture, ash, and sulfur content, while improvements are probable from combined measurements.

**PLANS FOR THE COMING YEAR** – The development of the MERC element meter and Btu measuring capability of monitor groups will be associated with the testing of the British Phase 3A ash monitor. The units will be compared for applicability, accuracy, and reliability of test coals.

## FINE COAL DESULFURIZATION AND RECOVERY TECHNOLOGY

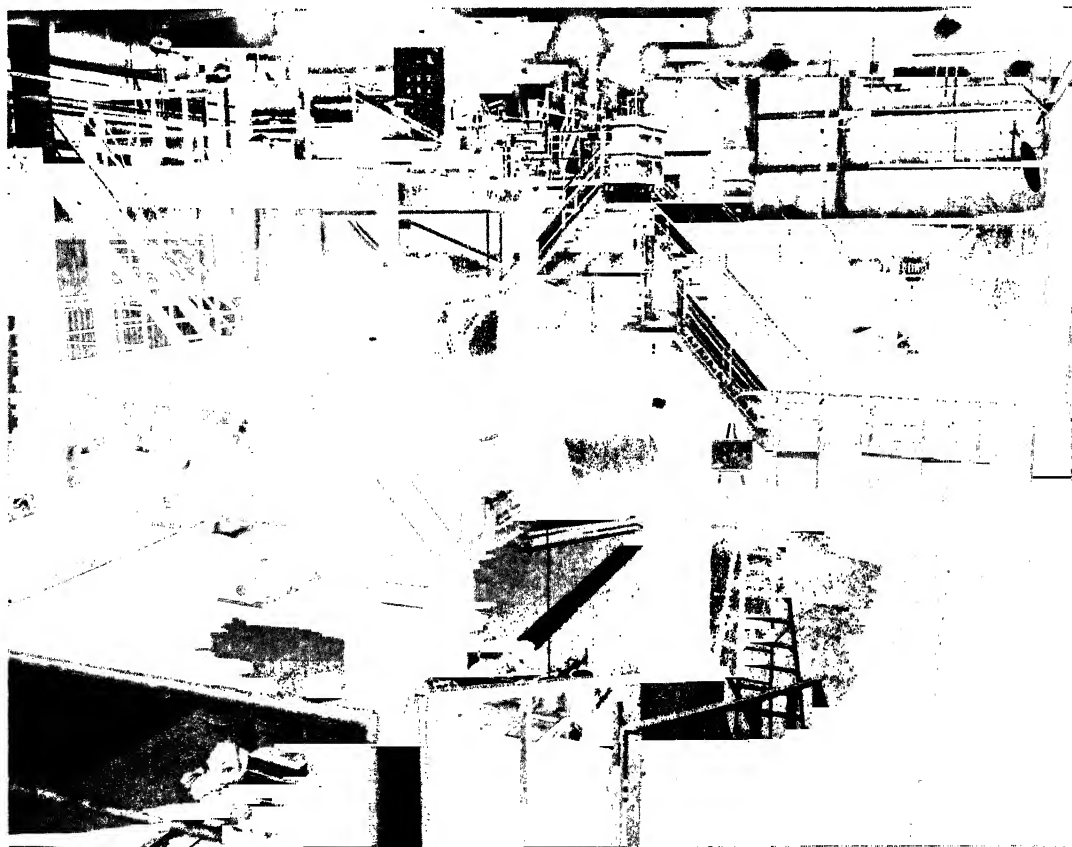
AMES LABORATORY

DOE - \$275,000

7/1/76 - Continuing

**OBJECTIVES** – The task is to develop and improve industrial methods for desulfurizing and recovering fine coal. These methods include froth flotation, oil agglomeration, pelletization, and chemical desulfurization.

**RECENT WORK AND ACCOMPLISHMENTS** – Two physical separation methods, froth flotation and selective oil agglomeration, and a chemical leaching method were developed further. In addition, preparations were made to develop the pelletization of fines because it can serve as a useful adjunct to these methods. As a result of numerous experiments to study various ways of improving the froth flotation and oil agglomeration methods of cleaning coal, the effectiveness of a new chemical pretreatment step was demonstrated. This step seems to alter the surface of the pyrite selectively so that a much better separation of coal and pyrite is obtained. Also, the effectiveness of a new chemical leaching process for removing both inorganic and organic sulfur from coal was improved through testing of various parameters such as temperature, pressure, concentration, and particle size. Construction was started on a continuous-flow, large-bench-scale system that will demonstrate froth flotation, oil agglomeration, and pelletization of coal fines and also the new chemical pretreatment step.



*Equipment Being Installed To Demonstrate Froth Flotation, Oil Agglomeration, and Pelletization at the Ames Laboratory Coal Preparation Plant*

**PLANS FOR THE COMING YEAR** – Laboratory development and optimization of the froth flotation and oil agglomeration methods of separation and the chemical pretreatment step for these methods will continue. Construction of the continuous-flow bench-scale system will be completed, and it will be placed in operation. Laboratory development and optimization of the chemical desulfurization process will also continue.

### CARBONIZATION

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$150,000

1974 - 1977

Principal Investigators - C. Ortuglio, J.G. Walters

**OBJECTIVES** – Investigations are being conducted in coal carbonization that will lead to more efficient utilization of premium coking coals while improving or eliminating environmental pollution associated with the process. One approach to pollution abatement is preheating and pipeline charging of the coal to the coke oven. The effect of preheating on the expansion properties of coals must be determined because excessive expansion during the coking cycle can cause costly oven damage.

**RECENT WORK AND ACCOMPLISHMENTS** – Expansion and contraction characteristics were determined for a number of coals typical of various ranks and types by subjecting them to preheat temperatures of 200°, 225°, and 250°C. Because coals are never coked as single entities but as blends of high- and low-volatile coals, the expansion characteristics of preheated blends were also investigated. Most high-volatile coals contract during carbonization but tend to contract less as preheat temperatures are increased. Pittsburgh bed coal has a normal contraction of 10.9 percent but when preheated to 200°C, this same coal contracts only 8.2 percent during carbonization. Corresponding values for preheat temperatures of 225° and 250°C are 6.7 percent and 2.8 percent, respectively. Pocahontas No. 2 bed coal, an excellent low-volatile coking coal, normally exhibits very strong expansion characteristics upon carbonization (19.2 percent) but upon preheating to 225°C, the expansion decreases to 4.6 percent. The decrease in expansion noted for the low-volatile coal combined with the decrease in contraction noted for the high-volatile coal tend to neutralize each other resulting in no change in expansion of blends of coals caused by preheating. This effect was noted for blends of various coals.

**PLANS FOR THE COMING YEAR** – The project has been terminated, and the results of the investigation will be published.

### CHEMICAL REMOVAL OF SULFUR FROM COAL (OXYDESULFURIZATION)

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$200,000

1971 - Continuing

Principal Investigators - S. Friedman, F. Steffgen, J. Ruether

**OBJECTIVES** – This project is to provide part of the laboratory process R&D necessary to develop the oxydesulfurization process to pilot plant operations and commercial demonstration. Oxydesulfurization is a method developed at PERC in which sulfur is selectively oxidized from coal in an aqueous slurry by treatment with air at elevated pressure and temperature. Removal of well

over 90 percent of pyritic sulfur and as much as 40 percent of organic sulfur is possible, transforming many high-sulfur coals to boiler fuel that does not require stack gas cleaning to meet air quality standards for sulfur oxides.

**RECENT WORK AND ACCOMPLISHMENTS** – All oxydesulfurization experiments were conducted in a 1-liter stirred batch autoclave, and a continuous flow of compressed air was passed through the reactor to maintain constant oxygen partial pressure. The effects of important process variables were studied such as temperature—150° to 200°C, pressure—500 to 1200 psig, stirring rate—300 to 900 rpm, reaction time, and coal particle size. A series of oxydesulfurization reactions conducted at the same conditions with two mesh sizes, 14 by 0 and 200 by 0, of seven different coals has shown the larger size consist desulfurizes somewhat more slowly, indicating a significant intraparticle diffusion resistance. Many different coals have now been screened for their response to a standard set of oxydesulfurization conditions. Typically, over 90 percent of pyritic sulfur and from 20 to 40 percent of organic sulfur are removed. Construction of a 10 to 40 kg/d unit for continuous processing of coal using a slurry bubble column reactor has been completed, and the unit is being tested prior to startup. Two economic analyses of the process by independent engineering consultant firms concluded that oxydesulfurization has excellent prospects for commercial development.

**PLANS FOR THE COMING YEAR** – The success of laboratory studies of the oxydesulfurization method and the favorable technical and economic review by Bechtel Corporation dictate the accelerated development of this process to pilot plant scale. Plans call for continuation of process variable studies in both batch and continuous flow units at PERC, but an overall program plan for commercialization is being developed that will encompass contracting laboratory and engineering process R&D and pilot plant (5 to 10 t/d) operations at various industrial and Government laboratories.

## CONTROL OF ASH FOULING

GRAND FORKS ENERGY RESEARCH CENTER

DOE - \$330,000

1976 - Continuing

**OBJECTIVES** – Reduced boiler efficiency and availability resulting from ash fouling has been the greatest single problem facing utilities burning low-rank Western coals, particularly those containing appreciable sodium in the ash. Planned changes in firing methods for reducing NO<sub>x</sub> emissions could aggravate this problem. This project evaluates the effects of coal characteristics and boiler operating conditions on the severity of fouling. Utilities use this information to work out partial solutions for new applications through suitable boiler design. Other remedial measures studied include selective mining, boiler load management, fuel additives, and coal cleaning.

United Power Association, confirmed the overriding importance of sodium content in determining the fouling tendency of North Dakota lignites. Combustion additives containing calcium, magnesium, or aluminum tested in the laboratory furnace reduced the strength of deposits but did not significantly effect the rate of deposit growth. A series of laboratory tests completed on geographically representative samples of Texas lignites, most of which contain high ash and low sodium, typically resulted in loosely bonded accumulations of ash that adhered to refractory but not to metal heat transfer surfaces; these deposits would not be expected to cause serious operating difficulties except in the presence of high-sodium content. Basic research employing mineral separation, x-ray diffraction, and microprobe examination was performed to characterize minerals and their spacial distribution in selected samples of North Dakota lignite and Montana and Wyoming subbituminous coals. Exploratory tests employing a new scanning and analyzing electron microscope and a heated-stage microscope discovered that many of the fly ash particles within ash deposits are composed of two or more layers that melt and volatilize at different temperatures.

**PLANS FOR THE COMING YEAR** — Additional tests will be performed in the 75-lb/hr laboratory furnace on coal fineness, excess air, furnace mixing, and ash elements auxiliary to sodium prior to publishing a major report of investigation on fouling. Direct furnace injection of sodium will be tested to determine if this practice can serve as a flue gas desulfurization technique without causing catastrophic fouling. A test series will be run on selected Eastern bituminous coals to establish a comparison with the fouling tendencies of the Western coals; tests will also be conducted on peat. Field tests on additives will be conducted in cooperation with utilities burning high-fouling coals, using background from past statistical studies as a control data base for comparison. Over-fire air will be further tested to determine if it can provide significant relief from fouling, as well as lowered  $\text{NO}_x$  emissions. Basic research initiatives will involve continued studies on coal mineral forms and deposits using the scanning and analyzing electron microscope (SEAM) and the heated-stage microscope. A major goal for SEAM is to develop and apply computer software for automated scanning. A study of fly ash formation from lignite will be carried out by SEAM of ash particles sampled from small pulverized-coal flames under a contract with Midwest Research Incorporated.

## COAL SLAG BASED GLASSES FOR HOT GAS CLEANUP

GENERAL ELECTRIC COMPANY

DOE - \$103,202

4/26/77 - 4/25/78

**OBJECTIVES** — This study program is seeking to demonstrate the technical and economic feasibility of using coal slag based glasses as a fluid to entrain particulate matter at high temperatures without significant carryover, which will permit direct utilization of the effluent from a coal combustor without cooling the gases to effect the cleanup. Emphasis will be on providing gas sufficiently free of particulates to permit turbine operation without undue corrosion or erosion. The critical importance of cleaning high-temperature high-pressure coal-derived combustion gases for use in combined-cycle gas turbine applications to improve overall system efficiency is well known and is being studied in several different approaches. In this one, the usually glassy nature of the particles is capitalized upon by providing a glass coated "sticky" surface, as well as baths, to trap and dissolve the particles which prevents them from ricocheting, fracturing, and becoming re-entrained. In addition to this primary benefit, successful accomplishment of the objective should provide more compact, easily handled waste products. The glassy form of the fly ash occupies only

20 to 25 percent of the volume of powdery fly ash and should have some economic value and considerable utility as a primary raw material or as at least an aggregate in building materials.

**RECENT WORK AND ACCOMPLISHMENTS** – Fly ash from Montana Rosebud, Pittsburgh No. 8, and a mixture of 70:30 Illinois No. 6 and dolomite have each been used, along with a standard soda lime silica and an amber container glass to perform viscosity and dissolution studies. Mixtures of the various fly ashes and glasses were prepared as small right cylinders that were heated at 1000° and 1100°C for 1 to 20 hours on a 30-degree inclined plane to ascertain their fluidity and chemical reactivity. Results indicate that particularly the amber container glass retains its fluidity with up to at least 15 percent additions of the Illinois 6/dolomite fly ash and that it will essentially completely dissolve up to 75 percent fly ash in as little time as about 20 hours. It is expected that even more fly ash can be dissolved in longer times. Simultaneously, work on particle collection has been performed in a clear-plastic labyrinth-type duct using glycerine at various low temperatures to simulate the eventual molten glass. Three variations in design of the duct have been made with the best performance involving the addition of a bubbler type cleaner that removes over 99 percent of all of the particles and essentially 100 percent of particles larger than 2 microns. Both the labyrinth and bubbler features are now being incorporated in a hot test duct in which the amber container glass will also be used to demonstrate the technical feasibility of this approach to hot gas cleanup. Based on these results, a preliminary conceptual design and economic analysis has been carried out using a Coal-Fired Combined-Cycle (CFCC) system. It is estimated that a “sticky wall” unit would be 25 percent less expensive than the granular bed filter and with certain assumptions, which are yet to be confirmed, might replace both the cyclones and granular bed filters at a significant savings in cost and complexity.

**PLANS FOR THE COMING YEAR** – The hot duct test apparatus being constructed at the mid-point of this program will be operated to demonstrate the technical feasibility of hot gas cleanup using coal slag based glasses and to gain somewhat more data for the design of a second-generation laboratory scale test unit.

## REGENERATIVE PROCESS FOR DESULFURIZATION OF HIGH-TEMPERATURE COMBUSTION AND FUEL GASES

BROOKHAVEN NATIONAL LABORATORY

DOE - \$357,000

1976 - Continuing

Principal Investigators - R.T. Yang, M. Steinberg

**OBJECTIVES** – Basic process chemistry information is required for the development of processes for the regeneration of limestone or lime-based sorbents used in the desulfurization of combustion and fuel gases in power production cycles. It will be applied to fluidized-bed combustion (FBC) of coal. To identify the existing problems in FBC and to provide solutions and improvements are essential parts of the program. Fundamental understanding of the kinetics and mechanisms of the reactions in FBC provides the building blocks upon which the process applications can be firmly established.

**RECENT WORK AND ACCOMPLISHMENTS** – The BNL Regenerative Process involves reacting  $\text{CaSO}_4$  and fly ash from FBC in a kiln-type reactor. A steady 5 percent  $\text{SO}_2$  regenerated stream has been obtained at 1000°C kiln temperature, and the regeneration of a 30 percent sulfated Greer lime

from Argonne's pilot FBC can be completed in about an hour. The reactivity of the regenerated stone is 30 percent higher than the raw stone. An  $\text{Fe}_2\text{O}_3$ -catalyzed sulfation process has been discovered and is under further development; 1 percent  $\text{Fe}_2\text{O}_3$  coated on the lime particle surface can increase not only the sulfation rate (about doubled) but also the capacity. Calcium silicates are being investigated for their sulfation and regeneration abilities. Most of the silicates (formed at about  $1060^\circ\text{C}$ ) possess about the same sulfation ability as Greer lime but their regeneration rates are about 5 to 10 times higher. Kinetics of sulfation and regeneration reactions are being studied in a 1- and 10-atm TGA, and a 1-inch quartz fluidized-bed sulfator. Emission and corrosion aspects of the NaCl-treated FBC have been studied quantitatively.

**PLANS FOR THE COMING YEAR** — Studies are being continued on the BNL Regenerative Process, the  $\text{Fe}_2\text{O}_3$ -catalyzed sulfation and regeneration processes, sulfation and regeneration of the mono- and di-calcium silicates, formation of the reactive calcium silicates, and the silica-supported regenerable (synthetic) sorbents. Fundamental understanding of the interplay of the chemical reaction and pore diffusion in these heterogeneous systems will also be continued. Three atmospheric TG systems, one pressurized TG, a 1-inch fluidized-bed sulfator and a 10-g rotary kiln are being used. A 2-in. batch combustor and a rotary kiln regenerator with a similar capacity will be built for further process studies as well as basic studies. Based on the information obtained, process design and analysis studies will be performed to determine the economics of the systems.

## **FIRESIDE FOULING AND CORROSION**

**MORGANTOWN ENERGY RESEARCH CENTER**

**DOE - \$251,000**

**7/69 - 9/78**

**OBJECTIVES** — Fireside fouling and corrosion problems in coal-fired boilers require an improved characterization of the coal/mineral interactions, during the combustion and heat transfer processes, to achieve better performance and availability of the boiler. Specific objectives involve the use of a redesigned plug-flow combustor to achieve better tractability of the degradation of coal minerals; and an evaluation of the deposition of volatile species of corrodants, and their interactions with surface materials.

**RECENT WORK AND ACCOMPLISHMENTS** — In-house work on the various components of the plug-flow combustor has been completed, and operational units are being tested prior to assembly. Contract work on the gas/solid sampling probe and pneumatic pyrometer has not been completed. Free energy calculations have been performed to determine the equilibrium state of sodium chlorine, sodium hydroxide, sodium sulfate, chlorine, atomic sodium and atomic chlorine in combustion atmospheres. In addition, the conditions under which these species can form condensed phases has been determined. The role of corrodants other than sodium sulfate (e.g.,  $\text{S}_2$  and Cl) has been documented in a report that reviews the physical and chemical basis of current hot corrosion theories, including gas phase corrosion, by ionic and radical species.

the MERC model for high-gradient magnetic separation. Development of the plug-flow combustor will continue with the expectation that it will provide species for these studies.

## DIRECT SAMPLING AND CHARACTERIZATION OF GASEOUS SPECIES

MIDWEST RESEARCH INSTITUTE

DOE - \$197,304

5/1/76 - 10/31/78

Principal Investigators - T.A. Milne, F.T. Greene

**OBJECTIVES** – This research program has the twin goals of identifying the gaseous alkali metal species that may play a role in fireside corrosion, and developing direct mass spectrometric probes for sampling such species from both idealized laboratory flames and small-scale coal combustors. A knowledge of the gaseous alkali metal species and their behavior during pulverized coal combustion should help elucidate the mechanism of fireside corrosion. A better understanding of this mechanism should assist in finding ways to reduce fireside corrosion, thereby permitting improved Garnot efficiencies and greater boiler availability.

**RECENT WORK AND ACCOMPLISHMENTS** – A molecular-beam mass-spectrometric sampling system for the study of minor species in coal dust-air flames was designed, constructed, and tested. Interferences caused by ionization by particulates and metastable species were eliminated, and the system capability was demonstrated by sampling alkali metal and other species as parts per million levels from coal dust-air flames. Limited attempts were also made to improve laboratory coal-dust feeder and burner designs.

**PLANS FOR THE COMING YEAR** – The principal effort will be directed toward the design, construction, and testing of a molecular-beam mass-spectrometric sampling probe compatible with the combustors at MERC. Work on the identification of alkali metal species in laboratory coal-dust flames will also be continued.

## FORMATION OF $\text{NO}_x$ AND OTHER PRODUCTS FROM CHEMICALLY BOUND NITROGEN

EXXON RESEARCH AND ENGINEERING COMPANY

DOE - \$533,350

9/30/77 - 11/30/79

**OBJECTIVES** – This research is to separate the process of coal particle combustion into a series of sequential stages (pyrolysis, gas phase combustion, and char burnout), and to carry out both theoretical and experimental studies on the formation of  $\text{NO}_x$  and other products from chemically bound nitrogen that occurs in these stages. The results of this program will provide insight into the fundamental processes of coal combustion, and they will be used to construct useful diagnostic models of the formation of  $\text{NO}_x$  from chemically bound nitrogen in coal combustion. The ultimate aim of the models is the optimization of coal burner design and operation for  $\text{NO}_x$  abatement.

**RECENT WORK AND ACCOMPLISHMENTS** – This program is in the startup phase. Diagnostic equipment used in studying the pyrolysis stage of coal particle combustion is being put into operation. Theoretical work on both pyrolysis and char burnout is being initiated. This program is a continuation of work that was funded under NSF RANN Grant No. AER 75-03964. In that work, the techniques used in the pyrolysis studies were developed as was an early pyrolysis model. Initial



results were obtained on the devolatilization of nitrogen from four coals and one oil shale. Quantities of major gaseous species devolatilized were also measured under simulated combustion pyrolysis conditions\*.

**PLANS FOR THE COMING YEAR** – The pyrolysis of coal particles under simulated combustion heating conditions will be studied in both inert and chemically reactive atmospheres. The total mass and total nitrogen evolution from the solid phase will be determined as a function of heating conditions as well as the nature of the major volatile species that are evolved (with emphasis upon the nitrogen species that are evolved). In parallel with this work, a theoretical model will be developed of the pyrolysis process that will be calibrated with the experimental data and then used to extrapolate that data to other conditions. This work will include a variety of coals that are broadly representative of available U.S. coal types. Work on the theoretical char-burnout model will also proceed as will the design of experiments to investigate the gas phase combustion of important nitrogenous pyrolysis products.

## STUDY OF CHEMICALLY REACTING TURBULENT FREE SHEAR LAYERS

AEROCHEM RESEARCH LABORATORIES, INC.

DOE - \$262,592

9/76 - 11/78

**OBJECTIVES** – Concentrations of chemically reacting species in a turbulent reactor are being measured. These measurements are needed to test theories of turbulent, chemically reacting systems, and as input to modeling of realistic combustors.

**RECENT WORK AND ACCOMPLISHMENTS** – A flow system has been designed and assembled. A laser photolysis/chemiluminescence technique for obtaining the required concentration measurements on a model chemical system has also been assembled. Required lasers and electro-optical detection equipment have been acquired and tested. The turbulent flow field comprises a turbulent jet of  $N_2$  containing  $O_3$  that exhausts into, mixes, and reacts with a nearly stationary surrounding mixture of  $NO$  in  $N_2$ . The reaction of  $O_3$  with  $NO$  to product  $NO_2$  is the model chemical reaction chosen for study. Extensive hot wire anemometry measurements have been made to characterize the fluid mechanics of the flow system. A number of concentration measurements have been made at various points in the flow field. Preliminary results indicate a high degree of skewness in reactant concentration pdfs at high-shear locations in the mixing layer, and near Gaussian pdfs at other points. These results are directly applicable to advanced efforts in reacting flow and combustor modeling.

**PLANS FOR THE COMING YEAR** – Work will include a more complete characterization of the velocity field, including measurement of the Reynolds stress and turbulence energy spectra at points throughout the field. Similar measurements will be made in axisymmetric jet flows with variations in input reactant concentration and flow rates. Concentration measurements in flows with different (e.g., two-dimensional) geometries or extension of the concentration measurement techniques to obtain concentration power spectra or velocity/concentration correlations will also be performed.

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\*Under subcontract with the University of Arizona.

## RATES AND MECHANISMS OF COMBUSTION OF PULVERIZED COALS

| SANDIA LABORATORIES

DOE - \$250,000

4/1/77 - Continuing

**OBJECTIVES** – The combustion properties of pulverized coals are being characterized so that combustion modifications to minimize the formation of fouling, slagging, and polluting species can be evaluated. Coal combustion parameters influencing the conversion of sulfur, fuel-bound nitrogen, and mineral matter to gas phase pollutants, or otherwise undesirable products, will be determined in a laboratory-scale research program. The development of efficient and environmentally acceptable coal combustion processes requires the ability to simultaneously predict pulverized coal burning rates and formation rates of fouling, slagging, corrosive, and polluting species.

**RECENT WORK AND ACCOMPLISHMENTS** – Coal combustion parameters are being examined in a system that permits the injection of well-characterized pulverized coal or char into a controlled laminar gaseous stream generated by a gas-fed burner. Modification of the burner and construction of a fluidized-bed coal feeder have been completed. The system is now undergoing calibration including measurement of gas stream properties and calibration of the coal feeder. Optical systems to permit instantaneous and high-speed cinematography of entrained reacting coal particles have been assembled and are being evaluated. Thus far, resolution to at least 1 micron is obtained for suspended particles. Equipment required for the detailed chemical and physical characterization of coals, chars, and gaseous reaction products has been assembled. This equipment, which is now undergoing calibration, includes an elemental (C, H, N) analyzer for solid samples and a gas chromatograph as well as instruments for determination of weight loss, moisture content, ash content, ASTM volatile matter, and petrographic composition of coal samples. Crushing and grinding equipment have been modified and 1000-gm batches of Pittsburgh seam coal have been pulverized and classified. Calibration of the equipment for coal is nearly complete. Technical assessment of pulverized coal combustion problems and research needs in actual boilers and advanced combustors is being conducted through site visits. Reviews have been completed with coal combustion scientists at GFERC, MERC, PERC, Riley Stoker, Combustion Engineering, AVCO, United Technologies, Exxon, Shell, Midwest Research Institute, MIT, University of Arizona, Brigham Young University, as well as the principal university, governmental, and industrial coal research laboratories in Australia and New Zealand.

**PLANS FOR THE COMING YEAR** – Using high-speed cinematography, measurements of coal particle ignition, devolatilization, and char burnup intervals will be attempted. The same technique will be used to obtain descriptions of particle swelling, shattering, and the like as functions of measured burner gas temperature and composition, and physical and chemical properties of the unreacted coals. A two-color pyrometer will be assembled and applied to the measurement of reacting coal-particle color temperatures. A quench probe will be constructed and used to extract partially reacted solid material to obtain coal and char particle weight loss during combustion. These data will be used for the elucidation of ash-forming mechanisms. An aerodynamic quench probe will be constructed and used for obtaining gas samples at various points in the coal combustion stream for the purpose of defining chemical reaction mechanisms and rates. Coals for these studies have been selected from the Penn State coal bank and classified; pulverized samples will be characterized physically and chemically. Measurements will include particle-size distribution, porosity, density, swelling index, and proximate and elemental analyses. The technical assessment of pulverized coal combustion research needs should be completed.

## INVESTIGATION OF FUELS CONTAINING COAL-OIL-WATER EMULSIONS

GERMANTOWN LABORATORIES, INC.

DOE - \$494,000

8/24/77 - Continuing

**OBJECTIVES** – This program is evaluating the combustion of coal-oil-water slurries prepared by five different commercial emulsifiers. The project includes evaluation of the slurry systems, flame characteristics, and boiler efficiency utilizing a 40-hp boiler system.

**RECENT WORK AND ACCOMPLISHMENTS** – The program started in September 1977 with the set up of several emulsification systems, evaluation equipment, and related accessories. Production of experimental slurries is in progress.

**PLANS FOR THE COMING YEAR** – The five commercial emulsifiers will be evaluated, and flame characteristics will be investigated.

## COAL FUELS COMBUSTION MECHANISMS

LAWRENCE BERKELEY LABORATORY

DOE - \$20,000

1976 - Continuing

Principal Investigator - R.F. Sawyer

**OBJECTIVES** – The increased utilization of coal through direct combustion and the substitution of coal-derived fuels for natural gas and petroleum fuels is certain to create difficulties related to incompatibility with existing combustion equipment and burning methods and to the increased production of pollutant species. This work is measuring combustion properties (burning rates, blowout conditions, fuel thermal diffusivity, and pollutant formation) important to the design of new combustion systems, control of combustion processes, and reduction of pollutant emissions.

**RECENT WORK AND ACCOMPLISHMENTS** – The opposed flow diffusion flame laboratory burner was demonstrated to be applicable to the well-controlled combustion of pressed pulverized coal and graphite. Burning rate and temperature profile measurements were obtained. A burning rate dependence on the 0.74 power of the oxygen concentration was measured. The effect of water content in the fuel on burning rate and ash buildup characteristics was observed. A study of the combustion characteristics of methanol/coal slurries in a furnace facility was completed. Combustion characteristics including flame shape, spectral radiation intensity, flow velocity, temperature, and species concentrations were measured locally within the furnace. Data on pure methanol and methanol with up to 5 percent pulverized coal added were collected. A model for the prediction of spectral radiation intensity from an inhomogeneous mixture of combustion products was developed.

**PLANS FOR THE COMING YEAR** – Work for the coming year will include extension of burning rate measurements to solvent refined coal. Gas phase composition profiles are sought to help resolve the relative importance of surface and gas phase reactions in the combustion of coal fuels. The production of nitrogen containing product species from fuel contained nitrogen will be measured.

## COMBUSTION CHEMISTRY OF LOW- AND INTERMEDIATE-BTU GASES

BATTELLE, COLUMBUS LABORATORIES

DOE - \$341,240

6/22/76 - 7/7/78

**OBJECTIVES** – The program will develop the primary combustion parameters needed for a unified description of flame stability of low- and intermediate-Btu gas mixtures. Basically, the study is aimed at defining the chemistry of CO-H<sub>2</sub> interactions in combustion, and at obtaining experimental data needed for the prediction of flame stabilities and flame lengths of low- and intermediate-Btu mixtures over a wide range of mixture compositions. The program encompasses flame kinetics studies, burning velocity, and flashback measurements and quenching distance studies on laboratory and field gas mixtures. The fundamental combustion parameters developed in this program will allow one to identify nonlinear interrelationships of CO-H<sub>2</sub> in practical gas systems, predict the burning characteristics of low- and intermediate-Btu gas mixtures, and resolve fuel interchangeability problems and composition fluctuation problems for improved burning of fuels generated from gasification processes.

**RECENT WORK AND ACCOMPLISHMENTS** – Task A, Flame Microstructure Studies, and Task B, Flame Property Studies, made up the major effort in 1977; Task C, Burning Velocity Equation Analysis, is essentially the culmination of the study and begins in early 1978. In Task A, binary fuel (CO-H<sub>2</sub>-CO-CH<sub>4</sub>, H<sub>2</sub>-CH) and ternary fuel (CO-H<sub>2</sub>-CH<sub>4</sub>) mixtures are stabilized on a laboratory flat flame burner and the flames are probed for temperature and for the composition of reactants and products. The primary objective of Task A is to define the rates of depletion of CO and H<sub>2</sub> relative to the rate of heat release. To date in Task A the flame microstructure studies of binary fuel (CO-H<sub>2</sub>) mixtures have been completed. Stoichiometric and excess air binary fuel flame mixtures have been probed at CO-H<sub>2</sub> ratios of 1:2, 1:1, and 2:1. Rates of CO, H<sub>2</sub>, and O<sub>2</sub> consumption and H<sub>2</sub>O and CO<sub>2</sub> production and rates of temperature rise have been developed for these flames. Preliminary application of the rate data to fundamental burning velocity equations yields predicted velocities within a factor of 2 of those determined experimentally. Microstructure studies of ternary fuel (CO-H<sub>2</sub>-CH<sub>4</sub>) mixtures have just begun. Simultaneously with this study, rate parameters most suitable for use in predicting burning velocities are being determined. Work is also continuing on improving the rate analysis techniques and physical property evaluations to increase the accuracy of the burning velocity predicted values. The primary objective of the Task B experiments is to supply the burning velocity,  $S$ , flashback velocity gradient,  $B$ , and quenching distance,  $\delta$ , data for the six CO-H<sub>2</sub> fuel mixtures examined in Task A. The data were obtained over a range of air-to-fuel ratios, using conventional techniques. A comparison of the flashback velocity gradient data for the low heating value fuels with data from an earlier study showed the results were compatible, and confirmed a discontinuity previously observed in the data as the H<sub>2</sub>/CO ratio increased. Two dimensionless groups based on the experimental data are being investigated, the group  $G \delta/S$  and the Reynolds number  $S \delta \rho/\mu$ . The latter is being considered in place of the Peclet number, assuming a constant Prandtl number. The group  $G \delta/S$  is quite often assumed to be a constant for hydrocarbon fuels. The data showed a linear increase with adiabatic flame temperature and with ratio of H<sub>2</sub> to H<sub>2</sub> plus CO. Analysis of the data presented in the Reynolds number form shows a lesser but similar variation of this dimensionless group with H<sub>2</sub>/(CO + H<sub>2</sub>), compared to  $G \delta/S$ . For the two different inert compositions, the value of the dimensionless group changes with the square root of the combustible portion of the fuel.

**PLANS FOR THE COMING YEAR** – The main objective of the next phase of the work is to expand the data base to include methane in the fuel mixtures. Remaining work in Tasks A and B involves the development of rate analyses of ternary gas mixtures and possibly low-Btu field gas mixtures plus completion of flame property measurements for the ternary gas mixtures. Specific attention will be directed to the correlation of  $G$ ,  $S$ , and  $\delta$  and the two dimensionless groups as a function of adiabatic flame temperature and product composition. These flame property data plus the global rate data will then be applied to the burning velocity equations to develop expressions for predicting the burning velocity of low- and intermediate-Btu field gases.

## **BENEFICIATION OF DRY PULVERIZED COAL BY HIGH-INTENSITY MAGNETIC SEPARATION**

OAK RIDGE NATIONAL LABORATORY

DOE - \$200,000

2/1/77 - Continuing

**OBJECTIVES** – This study is aimed at demonstrating the technical feasibility of removing pyrites, heavy metals, and other inorganic materials from dry pulverized coal using high-intensity magnets; developing a coal separability assay method specifically applicable to dry-coal magnetic separation, and exploring practical systems for carrying out dry-coal magnetic beneficiation commercially on a large scale. Coal beneficiation reduces the quantities of ash and sulfur dioxide produced during coal combustion. It supplements (or in some cases replaces) combustion and postcombustion control technologies such as fluid-bed combustors and flue gas desulfurization and particulate removal systems. Dry magnetic separation, in addition, offers the advantage of utilizing finer coals ( $< 60$  mesh), thus resulting in higher separation efficiency and increased Btu recovery without the need for expensive dewatering and drying. It also can be utilized directly in a pulverized coal-fired power plant, thus taking advantage of existing pulverizer and coal-air transport equipment.

**RECENT WORK AND ACCOMPLISHMENTS** – The separability of three different coals has been determined using the Frantz Isodynamic Separator as an assaying device. Pyrite and ash separability was determined at a Btu recovery of 90 to 95 percent for several size fractions. The Frantz Separator was confirmed as being a useful device for characterizing the separability of coals in a magnetic field. The technical feasibility of dry separation of pulverized coal was demonstrated successfully using a high-gradient magnetic separator (HGMS) in which the coal was fluidized so that it could be recirculated internally over the matrix elements. Separations exceeding those reported for coal slurries in identical separators were obtained for several representative coals. Separation efficiencies (percentage of impurities leaving with the reject stream) as high as 80 to 85 percent for pyrites and 55 percent for ash were obtained in the HGMS-fluidized-bed separator, with coal (Btu) recoveries of 80 to 85 percent. Separation efficiencies agreed with those obtained in the Frantz Isodynamic Separator. Several alternate approaches to continuous dry-coal magnetic-separation equipment designs were identified for further evaluation and development.

**PLANS FOR THE COMING YEAR** – A small experimental magnetic coal separation loop will be constructed and operated to obtain process information useful for the design of larger equipment, particularly for a continuous-type separation of the kind required for a large-size industrial application. A study of the separability of coal based on the float-sink method (relative specific gravity) versus that based on magnetic separation (relative magnetic susceptibility compared with specific gravity) will be carried out to develop methods for estimating expected performance of coals in a dry-coal magnetic separator.

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# **MATERIALS AND COMPONENTS**

## **PLANT MATERIALS FOR THE GASIFICATION OF COAL**

METAL PROPERTIES COUNCIL, INC.  
DOE - \$4,601,292; AGA - \$951,845  
1972 - Continuing

**OBJECTIVES** — This materials evaluation program will provide engineers with data on materials, both metals and ceramics, which is not now available, and which is required for the design of coal gasification plants. The program to achieve this objective includes both laboratory screening and plant exposure tests in order to determine, in a minimum of time, which available materials (metals and refractories) are suitable for service in gasifying units. The hostile environments which appear to be characteristic of proposed processes may be divided into three categories: gaseous, aqueous, and gaseous bearing particulate matter. Laboratory test programs are in progress in all of these areas. Successful candidate materials, based on these screening tests, are then being tested for their engineering properties under environmental conditions simulating those they will experience in service. These conditions include gaseous environment, temperature, and pressure. The properties being tested include tensile strength, yield strength, etc., from room temperature up to that temperature at which creep becomes a determining factor. Creep rupture characteristics are being obtained at higher temperatures, at present up to 1850°F, but with consideration that higher temperatures for testing may be desired later. Other properties being tested include fracture toughness and fatigue. In addition, the physical properties are being obtained. Success of this study will establish those materials which can withstand the hostile environments found in coal gasification plants, and will provide the data required for designing commercial plants using these materials.

**RECENT WORK AND ACCOMPLISHMENTS** — The program has been subdivided into a number of phases. Phase I involves laboratory screening tests for gaseous corrosion. Two special autoclaves were designed and built in 1973 and 1974 at IIT Research Institute under the guidance of the MPC committee. Fifty test runs, ranging up to a maximum of 5000 hours have been carried out in these autoclaves. Fifty-nine alloys, or specifically processed alloys, have been subjected to exposure in this equipment. Tests have been conducted at 1000 psi pressure, at temperatures of 900 to 1800°F. The gas environment consists of H<sub>2</sub>O, H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub> and H<sub>2</sub>S. The hydrogen sulfide has been varied over the range 0-1.0 percent. The tests have indicated the relative corrosion resistance of materials for service in coal gasification atmospheres. Some materials are unsuited for such service. On the basis of the Phase I tests, eight alloys were selected as the initial alloys to be extensively tested for their mechanical and physical properties under coal gasification plant operating conditions (see Phase V). Phase II involves pilot plant exposure tests. This phase is monitored by feasible, test racks containing samples of candidate metals or refractories are inserted in available locations in pilot plant units. The pilot plants in which these tests are conducted include HYGAS, CONOCO Coal, Synthane, BIGAS, Battelle, and Steam-Iron. This test program was started with the first insertion in the HYGAS plant in July 1974. Tests in the Battelle and Steam-Iron units began in 1977. Tests on the first four units will be completed this year. The remaining two plants will continue the tests until April 30, 1979. Thirty-four refractory materials were exposed in these tests, together with the alloys tested in Phase I. Phase III consists of a laboratory screening test program for aqueous corrosion in which conditions of exposure are similar to those expected in the quench systems of gasification plants. Temperatures are much lower in these portions of the plants than in the gasifier units, and materials are exposed to water containing those contaminants resulting from exposure to the gaseous environment. Four autoclaves at IIT Research Institute have been equipped

for aqueous corrosion tests which were initiated in 1974. Corrosion is experienced by immersed material, by material above the waterline, and at the air-liquid interface. Tests to date have established the relative usefulness of a number of materials. A set of predictive equations has been devised based on the test results. Phase IV was designed to supplement the gaseous corrosion tests of Phase I with the added effects of solid particulate matter expected to be entrained in the gas stream in gasification plants. Beginning in 1974, design and construction of special testing equipment to accomplish the objectives of Phase V was undertaken. A test unit was assembled which accomplishes this except for the 1000 psi pressure expected in some units is operational. Preliminary tests indicate severe corrosion-erosion on materials in contact with char particles. The tests were conducted with a specific char from a known source. Design was completed for units to operate under pressure, but work on two units was halted in 1976 because of lack of funds. The preliminary tests on the unpressurized unit indicate the possibility that tests on the pressurized units will be of great interest and importance. Phase V involves tests for mechanical and physical properties in gasification plant environments. As an initial step in obtaining the needed engineering design information, 10 alloys have been selected for extensive testing on the basis of test results from Phase I. The alloys will be tested for strength, fatigue, and toughness characteristics under the harsh environmental conditions of service, as well as the effects of long-time exposure to these conditions. Sophisticated test equipment has been designed and constructed. Multiple test autoclaves, fully computer controlled, have been designed and are under construction at Southwest Research Institute. Initial tests only have been made, and these indicate the need for much more information in these new fields.

**PLANS FOR THE COMING YEAR** – It is expected a modification to the existing contract will activate Phase IV during 1978. A pressurized test unit will be assembled. It has already been designed and the components are purchased. Tests exposing samples to erosion by particulate matter as well as to corrosion by hot gases under pressure will be conducted during 1978. Complete construction of the test stand will be accomplished in Phase V in 1978. Test equipment for low cycle fatigue tests will be developed in 1978. Creep, tension, and Charpy tests will be conducted in air, and in CGA, and after long-time exposure in CGA.

## **NEWSLETTER ON MATERIALS AND COMPONENTS IN FOSSIL ENERGY APPLICATIONS**

**BATTELLE, COLUMBUS LABORATORIES**

DOE - \$103,368

8/17/77 - 8/16/78

Principal Investigators – J.R. Schorr, D.J. Maykuth

**OBJECTIVES** – This project involves the preparation and distribution of a newsletter that serves as a medium for exchange of information and experiences pertinent to the use of materials and components among the communities interested in the development of fossil energy systems. This function is especially important where the scaleup from pilot to commercial facilities is occurring so rapidly.

**RECENT WORK AND ACCOMPLISHMENTS** – Preparation of the newsletter began in 1975 with publication on a quarterly basis. In the second year, publication was increased to a bimonthly rate. As of October 1, 1977, 12 newsletters have been issued consisting of 8 pages that are available at no charge to qualified individuals involved or interested in present or potential materials and components activities related to the development of fossil energy systems. Distribution has



increased from approximately 3000 to 3500 copies. A key newsletter feature is the reporting of recent failure experiences with equipment and materials. Much of this information is transmitted through the Failure Prevention Information Center that was established at the National Bureau of Standards as a part of DOE's Failure Analysis and Reporting Procedure. Within the present contract period, over a dozen such failure experiences have been described in eight different pilot plants and process development units. In most cases, the reported descriptions included recommendations for avoiding future problems or lessening the severity of their effects. Topical articles presented recently include, "The Necessity for Coatings in Fossil Energy Conversion Systems," "Materials Requirements for Process Heat Exchangers in the Application of Gas-Cooled Reactors for Coal Conversion," and "Filtration of Liquid Coal."

**PLANS FOR THE COMING YEAR** — The newsletter will serve as a medium of exchange of timely information and experiences pertinent to the use of materials and components. Publication will continue on a bimonthly basis, and the format and content will be modified and improved to reflect the comments and input received from the technical communities involved.

## MATERIALS RESEARCH FOR CLEAN UTILIZATION OF COAL

NATIONAL BUREAU OF STANDARDS

DOE - \$585,000

10/1/76 - Continuing

Principal Investigator - S. Schneider

**OBJECTIVES** — The NBS materials research project will develop equipment and test methods and evaluate the durability of alloys and refractories in coal gasification process environments; determine properties of alloys, refractories, slags, and atmospheres that strongly influence the performance and lifetime of materials; and collect, evaluate, and disseminate information on operating experience and component failures in coal conversion process plants.

**RECENT WORK AND ACCOMPLISHMENTS** — The susceptibility of various alloys to stress corrosion cracking in the elevated temperature and pressure environment existing in coal gasification systems is being evaluated by the constant strain rate test. Austenitic stainless steels (310, 310S, and 347), a ferritic stainless steel (446), Incoloy 800, and Inconel 671 have been tested. The results suggest the possibility of premature brittle failure of coal gasification plant components made of 310 stainless steel, Inconel 671, and Incoloy 800 alloy. Equipment both for mechanical properties testing and for x-ray diffraction analysis of refractory concretes in simulated gasification environments at temperatures and pressures up to 1000°C and 1000 psig has been constructed. Room-temperature strengths of both a high- and low-alumina refractory concrete have been determined after exposure to a high-temperature H<sub>2</sub>O-CO<sub>2</sub> environment. Calcium carbonate is formed on exposure to the H<sub>2</sub>O-CO<sub>2</sub> environment at the expense of the hydrated calcium-alumina phases. The room-temperature flexural strength after 90 hours shows little change. The low-alumina refractory concrete actually showed an increase in strength. The erosion behavior after hydrothermal exposure correlates with the strength behavior and seems to be controlled by the same microstructural properties. High-temperature high-pressure apparatus for measuring the viscosity of molten-slag compositions is nearing completion. A heater temperature of 1200°C at a pressure of 240 psig steam has been reached. Since steam apparently enhances the formation of crystalline phases, some of which are more refractory than the original supercooled liquid mixture, temperatures in excess of 1450°C will be needed. By redesigning the platinum heater, the necessary



temperature can be attained. Under the failure prevention information program, a data base of approximately 500 reported operating incidents from several coal conversion process plants has been developed. Information can be retrieved from the computer-based file and used for statistical analyses and report preparation. A summary of incidents according to failure mode, material, and process plant has been prepared.

**PLANS FOR THE COMING YEAR** – Mechanisms for embrittlement will be studied by examining the microstructure of test specimens. Additional specimen configurations and alternate test methods will be analyzed in a continuing effort to develop a convenient in situ method of testing. Use of the high-temperature high-pressure mechanical loader for measurements of the strength of refractory concretes will begin emphasizing hydrothermal effects that can drastically reduce service life and performance of refractories. Use of the x-ray pressure vessel will permit the phase composition of specimens to be determined under the same simulated conditions used for measurements of strength. Viscosity measurements on various coal slag compositions in a hydrothermal environment will begin. A second sampling system for the mass spectrometer-molecular beam apparatus will extend the pressure range of vaporization and chemical transport studies to 20 atm. The Failure Information Data Center will analyze the operating records of the CO<sub>2</sub> Acceptor Pilot Plant in addition to reports from other sources, and add them to the data base. Preparations have been made for establishing a Materials Properties Data Center.

## MECHANICAL RELIABILITY OF MONOLITHIC REFRACTORY LININGS

BABCOCK & WILCOX COMPANY  
DOE - \$951,755; Babcock & Wilcox - \$107,066  
7/1/76 - 6/30/78

**OBJECTIVES** – This work will determine if highly reliable, crack-free monolithic refractory lining configurations can be developed for coal gasification process vessels. This cracking occurs during the initial dry-out, heat-up, cool-down, and subsequent recycling of these linings. To accomplish this objective, a pressure vessel test fixture will be designed, constructed, and lined with typical coal gasification process vessel linings. Performance of the various lining design configurations will be monitored at different heat-up and cool-down conditions. Expected results include new or improved materials and design configurations, installation and operational procedures, a mathematical model which will allow accurate predictions of refractory lining performance, development of high-temperature strain gauge techniques, and development of NDT techniques such as acoustic emission. Thus the understanding of how cracking occurs in monolithic refractory linings (which are considered to be cheaper, easier to install, lighter in weight, and better thermal insulators than brick) and how to prevent it will markedly advance the technology of coal gasification and improve the chances that safe and successful operation of commercial size coal gasification process vessels will occur.

**RECENT WORK AND ACCOMPLISHMENTS** – A pressure vessel/test furnace, which will accommodate a 5 x 1 ft refractory concrete lining, and will heat this lining with electric resistant heating elements to 2000°F at heating rates up to 300°F/hr, was designed, built, and installed. The vessel is U-code stamped to operate at 250 psi pressure up to 650°F; the center section is designed to withstand pressures from a combination of gas and refractory/shell interaction pressures of approximately 500 psi. An analytical code (math model) has been developed to predict the strains and/or stresses in the refractory lining and in the vessel shell. This analytical code is also being used

to reduce and analyze the refractory lining and vessel shell strains measured during the lining tests. Refinements to this model are expected as the experimental results are compared to the predicted results. An acoustic emission monitoring technique has been developed which can detect the occurrence of major cracking events in the refractory lining. An effort is now underway to include real time analysis capability with this technique. This additional capability could permit heat-up rate to be controlled by the acoustic emissions being generated in the lining during a dry-out, heat-up, and cool-down cycle. To date, a total of six lining tests have been run. The first two tests were run in air at 1 atm on a dual component monolithic refractory lining configuration. In the first test, uncoated anchors were used while in the second, coated anchors were used. All other test conditions and design configurations were basically the same. From previous work done on panels of these same materials and configurations, it was anticipated that coated anchors would reduce or eliminate cracking of the lining. It was found, however, that both linings cracked after a 1200°F heat-up at 100°F/hour with holds at 200°, 400°, and 1000°F. Further heating to 2000° intensified the cracking. The crack pattern generally followed the anchor spacing in both linings but was more extensive in the first lining. Because of the reduced intensity of cracking in the second lining test, further work with coated anchors is planned.

Based on the analytical code predictions, the monolithic refractory linings were generally expected to expand on heat-up, creep at high temperature (>1000°F) and to contract and crack on cool-down. The cracking was caused by the development of large tensile stresses in the hot face of the lining which exceeded the tensile strength of the refractory. These stresses were due to thermal gradient and time and/or temperature dependent shrinkage and creep effects. This lining performance was expected to cause the vessel shell to go into a state of tension on heat-up and compression on cool-down. When this predicted vessel shell stress was compared to the experimental results obtained during the first test, an unexpected time dependent shrinkage effect was indicated. The substitution of a time dependent shrinkage law in the analytical code which produced the equivalent of 0.3 percent linear shrinkage in the whole lining enabled the predicted results to more nearly match the experimental results. These results coupled with the reduced intensity of cracking in the second lining compared to the first indicate that the coated anchors have reduced but not eliminated the refractory/anchor interactions. These test results also indicate, however, that the shrinkage of the refractory material at temperatures as low as 200° to 400°F must be reduced below the 0.2-0.4 percent linear levels presently considered acceptable for these materials. These findings are presently being factored into the plans for the remaining work on this contract.

**PLANS FOR THE COMING YEAR** — Primary emphasis will be placed on improvement and/or verification of the test techniques used during the lining tests, and determining the mathematical relationships of the shrinkage and pore pressure of the materials with time and temperature. Greater emphasis will be placed on the acoustic emission monitoring of the linings, and the math model will be refined and completed. Two additional linings will be tested for several cycles each, the results being used to update and improve the math model and acoustic emission monitoring equipment. The results will also be used to develop the refractory specifications, design, and installation guidelines and operating procedures.

## CORROSION/EROSION PROTECTION OF COAL GASIFICATION VESSELS

INTERNATIONAL HARVESTER

DOE - \$200,000

9/27/77 - 10/1/78

**OBJECTIVES** — This program is designed (1) to develop a coating system(s) suitable for protecting large internal components of coal gasifiers from corrosion at rates greater than  $500\mu/\text{year}$  (20 mils/year); (2) to provide sufficient data on the coating composition, structure, and deposition techniques such that a transfer of data to the general materials industry will occur; and (3) provide data on the properties of the coating/substrate suitable for use by equipment designers. Reliability of the internal components of coal gasifiers such as cyclones, diplegs, grid supports, and instrument probes are critical to the economic operation of gasification processes; however, their size, location, and installation sequence prevents the use of protective strategies employed in other parts of the plant such as cooled walls coated with thermal barriers of castable refractories. In the hot areas,  $700^{\circ}$  to  $980^{\circ}\text{C}$ , the environment is quite aggressive, and corrosion has been predicted to proceed at rates up to  $500\mu\text{m}/\text{year}$  for alloys 304 and 310, stainless steel and Incoloy 800. The basic coating approach will utilize the extensive background on the reaction sintering processes for growing  $\text{M}(\text{CoNi})\text{CrAl}$  and refractory metal silicide coatings of metal surfaces. A combination of the thermal spray application of materials like  $\text{MCrAlX}$  and a subsequent process step to promote coating densification and bond formation to the underlying alloy will result in a means of protecting internal coal gasifier components. The thermal spray techniques to be developed will be amenable to field repair of coated surfaces.

**RECENT WORK AND ACCOMPLISHMENTS** — Substrate materials and powder compositions that will be evaluated are on order. These eight coating compositions will be applied on three different substrate alloys (type 304 stainless steel, type 310 stainless steel, and Incoloy 800).

**PLANS FOR THE COMING YEAR** — The work plan outlined for this program will entail corrosion testing to be performed in a retort where coupons of coated alloys will be partly immersed in coal char under an atmosphere of flowing steam/hydrogen/carbon dioxide and carbon monoxide with minor amounts of hydrogen sulfide, methane, and ammonia at  $980^{\circ}\text{C}$  ( $1800^{\circ}\text{F}$ ). The initial eight coatings will be reduced to four based on corrosion screening tests and metallographic verification of coating integrity. The ductility of these four coatings will be evaluated with thermal cycling and bend tests to select two coatings for further mechanical property testing. After the secondary mechanical property testing, coated panels of selected coating systems shall be supplied to DOE for exposure by DOE facilities for 5000 hours at  $980^{\circ}\text{C}$  under a typical coal gasifier atmosphere but at a pressure of 6.9 mPa (1000 psi). Selection of coatings to be tested at DOE facilities will be based on data gathered in the coating process optimization and testing stages of the program. The choice of coating systems will be made with the concurrence of the DOE technical representative.

## WELD OVERLAY IN COAL GASIFICATION SYSTEMS

INTERNATIONAL NICKEL CO., INC.  
DOE - \$197,735; International Nickel - \$10,407  
8/15/77 - 5/15/79  
Principal Investigator - E. P. Sadowski

**OBJECTIVES** — This program will accomplish the development and evaluation of weld deposited overlays to provide resistance to corrosion-erosion in coal gasification atmospheres (CGA). Generally, high Cr alloys have been most effective in withstanding corrosion-erosion at high temperatures in CGA. The size of coal gasification plants and the economics involved produce a need for the conservation of alloying elements which are necessary for corrosion resistance, particularly Cr. One means of conservation to use composite metal-on-metal structures in which a high Cr corrosion resistant layer is deposited on a lower alloyed substrate. This can be accomplished by weld overlaying. A benefit of the overlaying approach is that it circumvents the difficulty of fabricating high Cr alloys. Another objective is to establish the effects of a 1000-hour exposure in CGA on the metallurgical stability of the overlay and substrate. This will be accomplished by tests conducted in the pre- and post-corrosion exposure conditions to gain data on the extent of corrosion, and changes in elemental distribution, microstructure, and mechanical properties.

**RECENT WORK AND ACCOMPLISHMENTS** — Three filler metals, AWS ER309, INCONEL Filler Metal 72 (44 percent Cr/bal. Ni), and an experimental 31 percent Cr/3 percent Al/15 percent Fe/Ni base alloy is being overlayed on substrates of lower alloy content. Single and double layers are being deposited on AISI 304 and 310 SS and INCOLOY alloy 800H. Three weld processes are being used. The weld processes are: submerged-arc, hot wire gas-tungsten arc, and gas-metal arc. The welding of AWS ER309 filler metal on Type 304 SS is complete.

**PLANS FOR THE COMING YEAR** — Welding with the INCONEL Filler Metal 72 and R139 will be completed. Concurrently, a corrosion test cell will be built. Upon completion of welding, portions of the weldments will be exposed in a CGA. Mechanical properties and elemental distribution will be determined on specimens in the pre-corrosion exposure condition.

## HIGH-TEMPERATURE BEHAVIOR OF STRUCTURAL ALLOYS IN COAL CONVERSION ENVIRONMENTS

BATTELLE, COLUMBUS LABORATORIES  
DOE - \$227,248  
5/1/76 - 2/28/79  
Principal Investigator - I. G. Wright

**OBJECTIVES** — This program will evaluate the corrosion behavior of iron- and nickel-base alloys in coal gasification-type atmospheres with a wide range of oxygen, sulfur, and carbon potentials, and to relate the corrosion behavior to available thermodynamic and kinetic information, with the aim of understanding the parameters controlling the corrosion. In addition, statistical correlations will be developed between the corrosion rates of iron- and nickel-base alloys in coal gasification-type atmospheres and the gas composition, temperature, and pressure, to allow recommendations for the most economical use of materials as a function of the conditions of a particular process.

**RECENT WORK AND ACCOMPLISHMENTS** – A statistical test design has been developed which consists basically of a complete factorial of all combinations of alloy variables (at three levels), temperature (at three levels) and oxygen potential (at three levels), which leads to 162 combination. A modified autoclave test rig has been assembled in which all combinations of alloy variables and temperature can be accommodated in each run, so that only 18 runs are required to complete the design. Four commercially available, though relatively simple alloys, having the desired range of chromium and nickel levels, are employed in this program: USS 18-18-2 (Fe-18Ni-19Cr-2Si), Incoloy 800 (Fe-31Ni-20Cr-0.4Si), AISI 310 (Fe-19Ni-25Cr-0.7Si), and Inconel 671 (52Ni-48Cr-0.2Si). Six basic gas mixtures have been chosen to represent the range of conditions encountered in the various gasification processes, and the equilibrium compositions have been calculated for the various conditions of temperature and pressure called for in the factorial design. These data points have been superimposed on Ellingham-Pourbaix thermodynamic diagrams showing the range of stability of the various components of the alloys, to give an indication of the phases which might be expected to form at equilibrium, and relationship of the corrosion morphologies to the experimental conditions. A 1000 hour, segmented corrosion run has been completed in which small specimens of the four alloys above, plus an alumina scale forming alloy GE-1541 (Fe-15Cr-4Al-1Y), were exposed to the potentially most corrosive gas mixture of the factorial design for various periods and then withdrawn and metallographically analyzed to determine the rates of corrosion. This run allowed the descriptive metallographic measurement technique usually employed in evaluating alloy corrosion to be adapted to the current alloys and conditions, and the results indicated that 1000 hours was an appropriate exposure time to allow a meaningful assessment of the corrosion behavior of these alloys. The metallographically assessed total corrosion of the alloys was assessed metallographically as a function of time and temperature in the simulated coal gasification atmosphere used (mole fractions at 1 atm, 25°F, 0.2014 CO, 0.1151 CO<sub>2</sub>, 0.2014 H<sub>2</sub>, 0.1720 H<sub>2</sub>O, 0.010 H<sub>2</sub>S, and 0.300 CH<sub>4</sub>) at 1000 psi. Inconel 671 exhibited the best corrosion resistance at all temperatures, while the alumina-forming alloy GE-1541 proved as good or better. Preoxidizing GE-1541 was probably beneficial, but the benefit was difficult to gauge because of internal oxide stringers associated with the preoxidation. In these two alloys, corrosion penetration was by chromium-rich sulfides. The temperature dependence of the corrosion of the remaining three alloys proved complicated, but in general, surface loss was the result of the formation of scales containing the sulfides of iron, nickel, and chromium, and penetration was by chromium-rich sulfides. The observed corrosion modes were mostly those expected from consideration of the thermodynamics of the various systems.

**PLANS FOR THE COMING YEAR** – The corrosion exposures called for in the statistical test matrix will be made for fixed exposure times of 1000 hours, using modified tensile specimens. In addition to the measurement of the extent of corrosion by the methods used above, these will allow changes in uniaxial tensile properties to be measured and used in the statistical analysis.

#### DATA RECORD ON HIGH-TEMPERATURE OXIDATION AND CORROSION

BATTELLE, COLUMBUS LABORATORIES  
DOE - \$18,000; EPRI - \$18,000  
2/1/76 - 3/31/78

**OBJECTIVES** – Data are being compiled and assembled on the high-temperature corrosion behavior of metals and alloys used in electricity-generating plants. The resultant handbook could serve as a source of data for analysis and prevention of systems corrosion problems, as a guide for

the selection of materials for new systems, and as a means for identifying data gaps. A comprehensive record of existing and available high-temperature corrosion data for relevant alloys will provide a valuable summary of operating experience and indicate the limits of available data for engineers concerned with failures in current processes or with process modification or new applications.

**RECENT WORK AND ACCOMPLISHMENTS** – Three main sources of information have been investigated: reports of work on Government contracts, technical and scientific literature, and private industry. The latter source is regarded as the most important, and a large number of U.S. and European organizations have been approached with good response, although the release of some information has been very involved and slow. Government-sponsored research data banks such as DDC, NTIS, DOE, MCIC, and NASA, have been searched using a list of key words, and reports of interest have been obtained. A literature search of journals was made back to 1970; then, earlier data were traced by reading pertinent articles and by using two existing collections of pre-1970 literature. Card files of collected data have been set up, and the task of organizing the information in a form suitable for presentation is underway. Sections dealing with basic data (thermodynamic and diffusion measurements), fluidized-bed combustors, oil-fired boilers, and coal-fired boilers have been completed and are in the process of review and modification.

**PLANS FOR THE COMING YEAR** – The remaining sections will be completed; after review, the data book will be assembled in a form suitable for publication.

## INSPECTION TECHNIQUES FOR WEAR- AND PROCESS-RESISTANT COATINGS

OAK RIDGE NATIONAL LABORATORY

DOE - \$200,000

1/1/76 - Continuing

Principal Investigators - R.W. McClung, G.W. Scott

**OBJECTIVES** – This project is developing nondestructive methods to inspect thin protective coatings on metals used in coal process equipment. The detection of existing failures is mandatory; the detection of precursory conditions that warn of incipient failure is highly desirable. Inspection techniques underlie quality control of component fabrication and quality assurance in operating plants. They can also assist in materials development and failure analysis.

**RECENT WORK AND ACCOMPLISHMENTS** – Work on ceramic and cermet coatings is being directed toward a smooth conclusion, and the shift to metallic coating work is in progress. Several artificial-defect specimens were designed and built: unbonds, thermal discontinuities, missing-coating areas, variable thickness, and artificially-induced cracks. A system to study x-ray fluorescence testing for coating thickness and homogeneity has been installed and partially calibrated. Transmission radiography of various specimen types has a demonstrated ability to detect cracks beneath the surface in 0.25-mm  $ZrO_2$  coatings on 3-mm Incoloy alloy 800 (I800) substrates. Some defects open to the surface can be imaged by backscatter radiography. Thermography successfully detected a 2.5-mm diameter thermal defect (a simulated nonbond) under a 0.25-mm  $ZrO_2$  coating. The 3.2-mm I800 substrate was heated by induction methods that are adaptable for field use. Success with surface inspection materials has been limited. Capacitance methods for ceramics yield ambiguous indications; they cannot distinguish between changes in the coating thickness, porosity variation, and changes in the liftoff of the probe. Ultrasonics has a demonstrated

ability to detect missing coating areas and unbonds from the substrate side of a specimen. An eddy-current instrument has been developed that simultaneously measures ceramic coating thickness, as spacing between the probe and substrate, and the conductivity of the substrate. Sensing the conductivity enables the instrument to compensate and avoid errors in thickness indications caused by conductivity changes. Effective conductivity and permeability have been measured on free-standing cermet and alloy coating layers. The ability to distinguish between controlled and measured areas of ceramic-coated and uncoated surfaces of I800 has been demonstrated by an immersion differential electrode capacitance measurement. This electrochemical effect has been known for many years; our attempt may be the first to use it as a nondestructive test method. Demonstration experiments with our arc-discharge probe system showed its ability to detect and map cracks in a ceramic coating. All planned experiments have been completed and reported.

**PLANS FOR THE COMING YEAR** – Inspection methods will be developed for 0.12 to 0.05-mm thick plasma-sprayed CoCrAlY coating deposits on I800 substrates. Priority will be given to inspection of as-sprayed coatings. Development may be extended to coatings that have been peened, sintered, or hot-pressed (these processes seal and densify the coatings to improve corrosion resistance). Inspection requirements and the methods that will be tested for feasibility are crack/hole detection: surface methods, eddy current, radiography; one-side-coating thickness measurement: eddy current, x-ray fluorescence, ultrasonics; unbond/delamination detection: thermal (imaging), ultrasonics, electric current (4-point probe).

## **FRACTURE TOUGHNESS OF CANDIDATE STEELS FOR PRESSURE VESSELS**

OAK RIDGE NATIONAL LABORATORY

DOE - \$100,000

10/1/76 - Continuing

Principal Investigator - D. A. Canonico

**OBJECTIVES** – This program is directed toward the characterization of the tensile and fracture toughness properties of the plain carbon and low alloy steels that will be employed in the fabrication of large thick-walled pressure vessels and their juxtaposed piping. The materials of interest include SA 516 Grade 70, SA 533 Grade B Class 1, SA 204 and SA 387 Grade 22 Class 2 steels. In addition, we have included A 543 Class 1 steel as a future candidate in the fabrication of thick-walled pressure vessels because of its higher strength.

**RECENT WORK AND ACCOMPLISHMENTS** – Fracture toughness properties were determined for 254-mm (10-in.) thick A 543 Class 1 steel plate using both precracked Charpy V-notch ( $PCC_V$ ) and 0.394T compact specimens (CS). Tests with the  $PCC_V$  specimen were made on material removed from the surface from  $\frac{1}{4}$ -thickness, and from mid-thickness locations. Tests using 0.394T CS were made with material from the  $\frac{1}{4}$ -thickness location. The  $PCC_V$  toughness values were calculated

thickness depths of thick steel plates have been measured during typical austenitize, quench, and temper heat treatments.

**PLANS FOR THE COMING YEAR** – Fracture toughness testing of the candidate steels will continue. In addition, studies will be started to determine the effect of aging in both air and H<sub>2</sub>-rich environments.

## TECHNIQUES FOR WELDING AND CLADDING CR-MO AND LOW-ALLOY STEELS

OAK RIDGE NATIONAL LABORATORY

DOE - \$100,000

7/1/76 - Continuing

Principal Investigators - D.P. Edmonds, J.J. Woodhouse, J.D. Hudson

**OBJECTIVES** – The designs of industrial coal gasifiers present a number of fabrication challenges. This program addresses two: cladding and field-welding technologies (ORNL now serves in an advisory capacity with respect to development of economic and reliable field-welding methods).

**RECENT WORK AND ACCOMPLISHMENTS** – Several major pressure vessel fabricators were surveyed as their experience with clad overlaying type 320 on low-alloy or carbon steels. None has produced claddings with type 320 on a production basis at high-deposition rates. Experimental work involves evaluating weld deposits made by the submerged arc and gas metal-arc processes for wide ranges of welding parameters. Although these studies are not complete, several important observations on the mechanism of deposition, conditions suitable for welding, and conditions that lead to cracking of deposits on carbon steel plate were made. Fissuring has been found in several of the higher dilution deposits made by the gas metal-arc and submerged-arc processes. For the gas metal-arc deposits, higher dilutions were obtained for lower travel speed, shorter electrode extensions, and higher currents. Also, the addition of small percentages of oxygen to pure argon shield gas greatly increases weld penetration and dilution. For the submerged-arc deposits greater dilutions were obtained at higher currents, faster travel speeds, and faster oscillations. The submerged-arc deposits were found to be much less susceptible to fissuring than similar gas metal-arc deposits. It is important that several of the conditions that were used produced deposits that did not crack. The test plates from this work will be used to determine the corrosion resistance of these type-320 overlays.

**PLANS FOR THE COMING YEAR** – Work is aimed at defining more clearly the range of welding conditions that minimizes defects in alloy 320 deposits. A follow-on part of the study is the evaluation of the potential use of a transition material (such as an Inconel-type weld metal) between the low-alloy steel or Cr-Mo steel base plate and the stainless-steel cladding. Some work to characterize the corrosion resistance of these deposits will be performed.

## WEAR RESISTANT ALLOYS FOR COAL HANDLING EQUIPMENT

LAWRENCE BERKELEY LABORATORY

DOE - \$225,000

1/1/77 - Continuing

**OBJECTIVES** – This program is aimed at developing wear-resistant alloys for coal transportation and fragmentation equipment. It is anticipated that large tonnages of coal will be mined, sized, transported, and handled because of the move towards utilizing fossil fuels for energy production.



**RECENT WORK AND ACCOMPLISHMENTS** – The inservice operating conditions of coal transport and fragmentation equipment were identified. It was concluded that various combinations of dry or liquid slurry abrasion, impact loading, and temperatures varying from ambient up to 1100°F can be encountered. A number of laboratory abrasion tests were identified as possible candidates for use as simulators of actual service conditions and to provide wear data for ranking of experimental alloys. A dry abrasive-wear tester was also designed, constructed, and calibrated. A number of steels were investigated, and work to date indicated that the low-alloy ultra-high-strength steels could be suitable for ambient temperature applications and secondary hardening steels would be useful for applications involving elevated temperatures.

**PLANS FOR THE COMING YEAR** – The abrasive-wear properties of the developmental alloys will be determined using different testers based on the application for which the steel is being considered. The metallurgical characterization of the experimental steels will be completed. A wear tester to simulate 3-body wear at ambient and elevated temperatures will be constructed and used for ranking of the experimental steels.

## LOW ALLOY STEELS FOR THICK WALL PRESSURE VESSELS

LAWRENCE BERKELEY LABORATORY  
DOE - \$160,000  
10/1/76 - Continuing\*

**OBJECTIVES** – Work on this project aims to adapt or develop low alloy steels that can be field fabricated into large diameter, thick wall pressure vessels for coal gasification systems. The work centers about developmental studies on commercially available steels and on new alloy steel systems to meet this goal. The successful completion of this alloy development program will produce better steels for thick section pressure vessels. These steels will have sufficient fabrication “forgiveness” to allow on-site construction of containment vessels requiring limited post-weld heat treatments. The steels will have improved environmental resistance and will have adequate strength and toughness in thick section to insure reliable operation of large reaction vessels.

**RECENT WORK AND ACCOMPLISHMENTS** – Research during the first year has been directed toward five program tasks: (1) determining alloy design criteria, (2) developing methods of simulating thick plate material in the laboratory, (3) characterizing commercial steels in thick section, (4) modifying existing steels to achieve desired properties, and (5) developing new steels better suited to thick wall application. The first three tasks have been completed successfully and have been used as the basis for developing modified commercial steels with better properties. Two alloy steel systems, Mn-Mo-Ni steels and Cr-Mo steels, have served as the basis for the research. The Mn-Mo-Ni steels have been alloyed for improved strength, toughness, and environmental resistance. Three new alloys have been selected for thorough property investigation. The Cr-Mo steels have been alloyed for increased hardenability. Uniform thick section microstructures have been achieved leading to improved strength and toughness.

**PLANS FOR THE COMING YEAR** – Thorough characterizations of promising modified commercial steels will be undertaken. This characterization will entail mechanical property

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\*This work is continuing on the campus of the University of California, under the supervision of the University of California, Berkeley Campus Research Office.

assessment in different thicknesses and heat treatments. Environmental resistance and weldability will also be evaluated. New steels, relying on eutectoid decomposition for strength, will also be evaluated during the next year.

## HIGH-TEMPERATURE CORROSION RESISTANT MATERIALS

LAWRENCE BERKELEY LABORATORY

DOE - \$30,000

10/1/76 - 9/30/77

**OBJECTIVES** – This program was to develop an alloy for applications in coal gasifier internals (viz. cyclones, diplegs, etc.) with acceptable corrosion resistance and mechanical properties. The typical environments encountered in coal gasifier (high Btu) cause severe sulfidation problems. Also since the temperatures were expected to be high, there is a need for mechanical integrity. It has been shown that existing commercial alloys such as 310SS do not stand up to these environments. Hence there is a need to develop new alloys for high-temperature sulfidation resistance.

**RECENT WORK AND ACCOMPLISHMENTS** – Work being done concurrently at the Lockheed Palo Alto Research Laboratory and at the University of California, Berkeley, has shown that acceptable corrosion resistance can be attained in Fe-Cr-Al type alloys, where the amount of Cr is kept to as low a level as possible with the Al being a partial substitute for Cr. However, these alloys lacked strength at elevated temperatures. The alloy base was used in the present study to improve strength through intermetallic compound precipitation. This was achieved by making alloy additions of elements such as Ta, W, and Hf to obtain precipitation hardening.

**PLANS FOR THE COMING YEAR** – Although no contract money is available for the coming year, metallurgical characterization of the developmental alloys is planned as support work on a continuing contract between Lockheed Palo Alto Research Laboratory and the University of California, Berkeley.

## FEASIBILITY STUDY OF PRESTRESSED CONCRETE PRESSURE VESSELS

OAK RIDGE NATIONAL LABORATORY

DOE - \$46,000

2/1/76 - 2/1/77

Principal Investigator - W. L. Greenstreet

**OBJECTIVES** – The potential use of prestressed concrete pressure vessels (PCPV) for commercial-sized coal conversion systems was to be investigated. Conceptual designs of concrete pressure vessel and liner combinations for gasifiers in commercial-sized systems were to be developed and studied as vehicles for assessment and guidance. Major problem areas were to be identified, and test programs for feature and concept demonstration were to be defined and outlined. Prestressed concrete pressure vessels (PCPVs) offer advantages of direct suitability for field erection, ease of fabrication, nonrestrictive limitations on size, use of relatively inexpensive materials, and inherent structural safety. Therefore, the use of PCPVs in applications where large, heavy-walled steel vessels are required merits strong consideration, especially for large gasifier vessels in commercial-sized plants for high-Btu gas production.

**RECENT WORK AND ACCOMPLISHMENTS** — Gasifier vessels for commercial-sized HYGAS and Synthane coal conversion plants were selected for study. Conceptual design information for the gasifier vessels was obtained from C.F. Braun Company; the vessel proposed in the HYGAS case has a maximum inside diameter of 9.75 m (32 ft) and a height of 73 m (240 ft). The design temperature is 589K, (600°F) and the design pressure is 8.96 MPa (1300 psig). For the Synthane plant, the vessel inside diameter is again 9.75 m (32 ft) and the height is 33.5 m (110 ft). The design temperature and pressure in this case are 616K (650°F) and 7.41 MPa (1075 psig). Conceptual designs for both the HYGAS and the Synthane PCPVs were developed through consideration of the major elements in each. Top and bottom head and side openings were designed to meet process requirements and to provide access to each of the separate regions in a given vessel. Access is required for equipment and internal structural component installation, inspection, and removal, as well as for operational reasons. Top head access openings having inside diameters of 3.15 m (10 ft, 4 in.) for the HYGAS vessel and 2.74 m (9 ft) for the Synthane vessel were provided. Each of the major elements of the PCPVs were considered in detail in developing the conceptual designs. These major elements include: (1) the high-strength concrete and conventional steel reinforcing bars, (2) post-tensioned prestressing steel, (3) top head closure plug, (4) thin steel pressure retaining liner for the vessel and penetrations, (5) thermal barrier system (insulating and cooling system), and (6) an insulating system for the exterior surface. The concrete of the vessel is protected from the high process temperatures, 982°C (1800°F) and above, by layers of refractories inside the pressure retaining steel liner, insulating concrete between the liner and the structural concrete, and water cooling circuit tubes at the inner surface of the structural concrete. The vessel internal structural members were not considered, except as they would interact with the vessel. Base supports for the vessels were included in the scope of the study, but foundation systems were not.

On the basis of the conceptual designs developed, four areas were identified which require test evaluation and demonstration. Two of these areas are associated with performance demonstration, which is closely tied to feasibility, while the remaining two are for demonstrating structural adequacy of the designs through model tests as required by the applicable structural design code. A cost comparison was made between steel and concrete vessels for the HYGAS gasifier. The gasifier size was for a two-train plant with a capacity of  $500 \times 10^9$  Btu/d, and essentially no cost differential was found. The overall study has shown that PCPVs are potentially (both technically and economically) feasible for gasifier applications. Since the requirements for gasifier vessels are probably the most stringent, it is expected that other uses in coal conversion systems could be more readily accommodated.

**PLANS FOR THE COMING YEAR** — The feasibility study was completed and a final report published. Further work is not funded at present.

## **REFRACTORY LININGS FOR COAL GASIFICATION PROCESS EQUIPMENT**

U.S. DEPARTMENT OF THE INTERIOR, BUREAU OF MINES  
TUSCALOOSA METALLURGY RESEARCH CENTER  
DOE - \$225,000  
1974 - Continuing

loss from high-silica refractories in order to predict possible silica fouling of equipment downstream from the gasifier, and (3) studying the effect of alkali vapors on refractory liner materials. Refractory linings in second-generation coal gasifier reactors must perform in a hostile environment rich in steam, hydrogen, carbon dioxide, and carbon monoxide at high temperatures and pressures. Gasifier reactor linings serve as insulation to reduce heat losses, to lower metal pressure vessel shell temperatures, and to protect the metal reactor shell from erosion by moving coal and ash particles. Liner refractories with the ability to withstand long-term service between repairs or replacement must be identified, and improved refractories must be designed to assure the success of substitute natural gas production from coal.

**RECENT WORK AND ACCOMPLISHMENTS** — At reactor lining hot-face temperatures, steam and hydrogen were found to be the most active components of the high-Btu coal gasifier atmosphere. Hydrogen was found to be less active toward calcium aluminate-bonded refractory concretes than steam, but it produced the same trends in strength changes. Calcium aluminate-bonded intermediate-alumina (high-silica) concrete strength was improved significantly by exposure to pure steam, pure hydrogen, and mixtures containing these gases, while calcium aluminate-bonded high-alumina (low-silica) concretes were attacked by these two pure gases and gas mixtures containing them. Sixty percent alumina brick strength was increased by exposure to high-pressure steam. Pure hydrogen, at high pressures and high hot-face temperatures, did not adversely affect 90 percent alumina brick strength, but did significantly lower that of 45 percent alumina brick. Pure hydrogen atmospheres were found to produce a drastic reduction in the phosphorous content of a phosphate-bonded ramming mix, although apparently without serious effect on refractory strength. High-pressure carbon monoxide atmospheres were observed to produce moderate strength losses in low-alumina (high-iron) brick at 1100°C, but high-Btu gasifier atmospheres (with hydrogen sulfide present) containing appreciable carbon monoxide did not have an adverse effect. Pure carbon dioxide atmospheres were found to be detrimental to 45 percent alumina concretes, but a typical high-Btu gasifier atmosphere containing appreciable carbon dioxide drastically improved low-alumina concrete strength.

The observed early deterioration in high-alumina calcium aluminate-bonded concrete properties under nonslagging high-Btu coal gasifier hot-face conditions ceased by 160 hours, while there was a continued slow deterioration in high-alumina phosphate-bonded concrete properties during an entire 1000 hour exposure. Concretes showing an improvement in properties (45 and 55 percent alumina calcium aluminate-bonded concretes) up to 160 hours generally showed continued improvement between 160 and 1000 hours exposure to a typical high-Btu gasifier atmosphere. Silicon carbide refractories were destroyed by exposure to this atmosphere. Based on the results obtained thus far, an optimum hot-face lining could consist of an intermediate (45-60 percent) alumina concrete and/or high (but not necessarily more than 90 percent) alumina brick. Phosphate-bonded ramming mixes could be used as patching material. Silicon carbide should never be used. Rapid catastrophic failure of a well-designed nonslagging high-Btu coal-gasifier refractory liner, made from carefully selected commercially available materials, due to hot-face gaseous corrosion is not likely. The extra cost of the use of refractory materials of very high alumina content is not justified, and in some cases would result in poorer performance than the use of lower alumina materials.

**PLANS FOR THE COMING YEAR** — Evaluation of commercially available candidate liner materials will be continued by making chemical and physical property measurements before and

after extended exposure to high-pressure coal gasifier reactor atmospheres. Emphasis will be placed on exposing lower alumina, less expensive materials to low-Btu gases at high temperatures, and to high-Btu gases at lower temperatures, in order to obtain data on the complete range of process conditions. The effect of silica content on the steam resistance of high-alumina concretes will be measured in order to identify the mechanism by which the presence of silica retards steam attack. Silica loss from promising high-silica candidate refractory liner materials will be measured in order to identify and eliminate from consideration, refractories that could add appreciably to silica fouling of heat exchange equipment downstream from the gasifier reactor. The rate of alkali metal oxide vapor attack (in the presence of high-pressure gasifier atmospheres) on promising candidate liner materials will be determined in order to predict the effect of alkali metal oxide vapor on linings of reactors containing high-alkali coals.

## SULFIDATION RESISTANT ALLOY FOR COAL GASIFICATION SERVICE

LOCKHEED PALO ALTO RESEARCH LABORATORIES

DOE - \$298,431

5/25/77 - 7/23/77

**OBJECTIVES** — This program aims to design a high temperature alloy that will have improved resistance to attack in high sulfur coal gasification atmospheres compared with commercially available stainless steels and super-alloys. The specific goal is to design an alloy having a penetration rate of less than 20 mils/year at 1800°F in gasification atmospheres with 1 percent H<sub>2</sub>S. A further goal is to design an alloy low in chromium and free of nickel with good producibility and fabricability. The results of this study have considerable significance with respect to the utilization of coal as an alternate energy source. The timely availability of an alloy with adequate resistance to high sulfur coal conversion atmospheres will impact on process selection, plant design, operating conditions, and reliability. In addition, the availability of a low-Cr, Ni-free alloy will reduce dependency on steels containing large amounts of critical alloy elements, and will provide a low-cost nonstrategic material for use in energy conversion systems.

**RECENT WORK AND ACCOMPLISHMENTS** — Commercial high temperature alloys rely on the selective oxidation of chromium to form an oxide scale that will protect the alloy from attack by sulfur and/or carbon. It has been found that the ability of a chromic oxide scale to block the reaction with sulfur is a function of the steam and hydrogen sulfide content of the gas in which the scale is formed. The scale becomes less protective as the H<sub>2</sub>O content decreases or the H<sub>2</sub>S content increases. A limiting condition beyond which a liquid sulfide slag forms on the surface has been defined. This boundary lies in the center of the gas composition range for advanced coal gasification processes. Alloys like 310SS are subject to accelerated attack and failure by sulfidation if gas compositions approach or cross this boundary. The approach to design an improved alloy in this program was to add sufficient aluminum to steel to form a protective scale of aluminum oxide under conditions where an adequately protective chromic oxide scale cannot be formed. It was found that the alloy must contain both Al and Cr in balanced proportions in order to form protective alumina scales in gasifier atmospheres. The addition of 2-3 percent Al is adequate in steels with over 15 percent Cr, but the required Al addition increases as the Cr content decreases. Tests have shown that a minimum of 4.5 percent Al is needed in steels with 16-20 percent Cr to provide long-term reliable protection from sulfidation. An Fe-5Al-18Cr alloy resists attack in gases with 1-2 percent H<sub>2</sub>S and 10-40 percent H<sub>2</sub>O at 1500° to 1800°F. No internal or external sulfidation was detected after 1000 hours at 1800°F. The oxide scale formed on these alloys spalls

on cooling to room temperature. The resistance to attack is degraded by cyclic heating and cooling, and the composition must be modified to stabilize the alumina scale. It has been found that the addition of 0.5 percent Y or 1 percent Hf produces an adherent alumina scale. No oxide spalling or internal sulfidation was noted after ten 100-hour exposure cycles at 1800°F.

The alloy has been balanced in composition to provide the best combination of sulfidation resistance and mechanical behavior. The steel is embrittled on slow cooling if it contains more than 6 percent Al. Steels with over 20 percent Cr also will be subject to embrittlement by the formation of sigma phase. Corrosion studies revealed that steels with less than 5 percent Al and less than 17 percent Cr had marginal resistance to sulfidation and a useful life that did not extend much beyond 1000 hours. The addition of 1-2Mo could be used to strengthen the alloy without degrading sulfidation resistance or mechanical behavior. Manganese was found to be an undesirable impurity, and alloys with over 0.5 percent Mn had poor resistance to sulfidation. Hafnium was found to be a preferred addition compared with yttrium to stabilize the oxide. Hafnium has a more reproducible effect on scale adherence with less of an undesirable effect on microstructure and mechanical properties. The addition of 1 percent Hf gave optimum results. The use of Si instead of Al to develop more protective oxide scales on steel also was investigated. An Fe-17Cr-3Si developed a duplex scale that was very resistant to penetration by sulfur. A comparison of the behavior of Fe-Cr-Al-Mo-Hf and Fe-Cr-Si alloys with that of commercial stainless steels and Ni-Cr alloys was made. The oxide scale on the Fe-Cr-Si alloy spalls on cooling, and attempts to modify the alloy for scale adhesion were not successful. It has a useful life about 1000 hours. The Fe-Cr-Al-Mo-Hf alloy shows no indication of wear-out at 1000 hours and is concluded to have the best potential for further development. The optimum composition range for the improved alloy is 18-19Cr, 5-6Al, 1-2Mo, 0.8-1.2Hf, bal. Fe. This material meets or exceeds all program goals except those relating to high-temperature strength. The alloy has 20 percent strength of commercial austenitic stainless steels at 1600-1800°F and must be used in heavy sections under stressed conditions. The alloy is considered to have good potential as a surface coating or cladding material and could be a low-cost nonstrategic replacement for the currently used Ni-50Cr alloy (IN671). It has been tested under pressurized conditions and shows negligible corrosion after 1000 hours at 1800°F in a gasifier atmosphere with 1 percent H<sub>2</sub>S and 39 percent H<sub>2</sub>O at 1000 psi (68 atm) pressure.

**PLANS FOR THE COMING YEAR** – Four 50-lb ingots of alloys in preferred composition range have been produced for more detailed study of corrosion and mechanical behavior. The alloys will be fully characterized with respect to tensile, stress rupture, and impact strengths over a broad temperature range. The effect of 1000-hour exposure to gasification environments on mechanical behavior will be studied. The limits of gas composition for protective scale formation will be determined as a function of temperature from 1200 to 1800°F and long-term protective capability to 4000 hours will be assessed. Recommendations will be made for future development, scale-up to production, and utilization.

## FORMATION OF PROTECTIVE LAYERS ON ALLOYS USED IN COAL GASIFICATION ENVIRONMENTS

SANDIA LABORATORIES

DOE - \$160,000

2/1/76 - Continuing

**OBJECTIVES** – The cost and commercial acceptability of converting coal to a high-Btu combustible gas will be significantly improved through the use of relatively inexpensive, but highly compatible materials which allow efficient coal gasification processes to be utilized for long times. Present commercially available alloys have very marginal capabilities to survive for useful times at temperatures of 1000°C in typical coal gasification atmospheres. The general objective of this work is to develop metal alloys which have long-time compatibility with high-temperature high-pressure sulfur-rich gases. The specific objective is to modify the best commercial iron-based and nickel-based alloy to achieve substantial improvement in the life of the alloy. These modifications shall not alter the alloy's established melt practices nor degrade the mechanical and fabrication properties. To aid in accomplishing these objectives, a laboratory capability is to be established which simulates the high-temperature high-pressure complex gas environments typically found in coal gasification pilot plants.

**RECENT WORK AND ACCOMPLISHMENTS** – Two test facilities have been designed and fabricated to varying degrees of completeness; a multi-furnace ambient pressure facility and a high-pressure cell. The primary purpose of the furnaces operating at ambient pressure is to provide a facility where candidate alloys are studied in a wide range of environments. The most promising candidates will be subsequently evaluated in the high-pressure test cell in a six component ( $\text{H}_2\text{S}$ ,  $\text{H}_2\text{O}$ ,  $\text{H}_2$ ,  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{CH}_4$ ) gas mixture at 1000 psi and 1000°C. During the construction of the test cell, extensive tests have been conducted in the ambient pressure facility in a less complex mixture of ( $\text{H}_2\text{S}$ ,  $\text{H}_2\text{O}$ ,  $\text{H}_2$ ) but at  $\text{P}_{\text{S}_2}/\text{P}_{\text{O}_2}$  ratios which vigorously corrode the best commercial alloys. Initial efforts concentrated on improving the compatibility of two alloys, Ni-30Cr and Type 310 (Fe-20Ni-25Cr) stainless steel. The alloy modifications consisted of variations in Ti, Al, Mo, and Mn additions. Short-time (24-100 hour) ambient pressure tests at 1000°C demonstrated the excellent performance of Ni-30Cr-3Al, Ni-30Cr-4Ti, and 310-3Ti. The unmodified alloys failed in these tests due to both melting of Fe and Ni sulfides as well as extensive internal sulfide penetration. Longer duration tests involving cyclic conditions demonstrated the superiority of the Ti modified alloys when contrasted to those modified with Al. In Ni-30Cr-4Al the very protective mixed  $\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$  film, was unable to remain adherent during the temperature cycling. In contrast, the very adherent  $\text{Cr}_2\text{O}_3/\text{TiO}_2$  complex oxide which formed on the Ti modified alloys was unaffected by the cyclic conditions. These results were successfully repeated in material in the as-welded condition. The room and elevated temperature mechanical properties of the Ti modified alloys are very similar in all respects to the unmodified compositions. In its first long-time test run, the high pressure cell was operated for 200 hours at 1500°F and 1000 psi. Design modifications are being

standard  $P_{S_2}/P_{O_2}$  gas composition will be studied. Two basic studies will be started: The mechanism by which Ti improves the compatibility and the mechanism of eventual breakdown of the protection as the corrosive nature of the atmosphere is increased.

## COAL LIQUEFACTION SYSTEMS FAILURE PREVENTION AND ANALYSIS

OAK RIDGE NATIONAL LABORATORY

DOE - \$152,000

1975 - Continuing

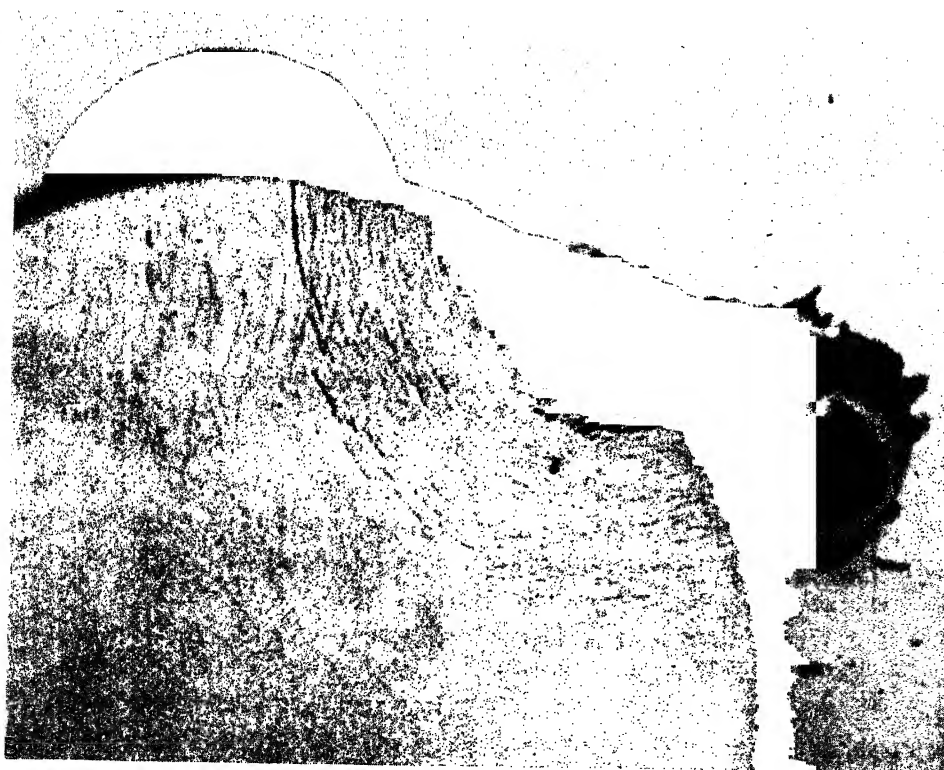
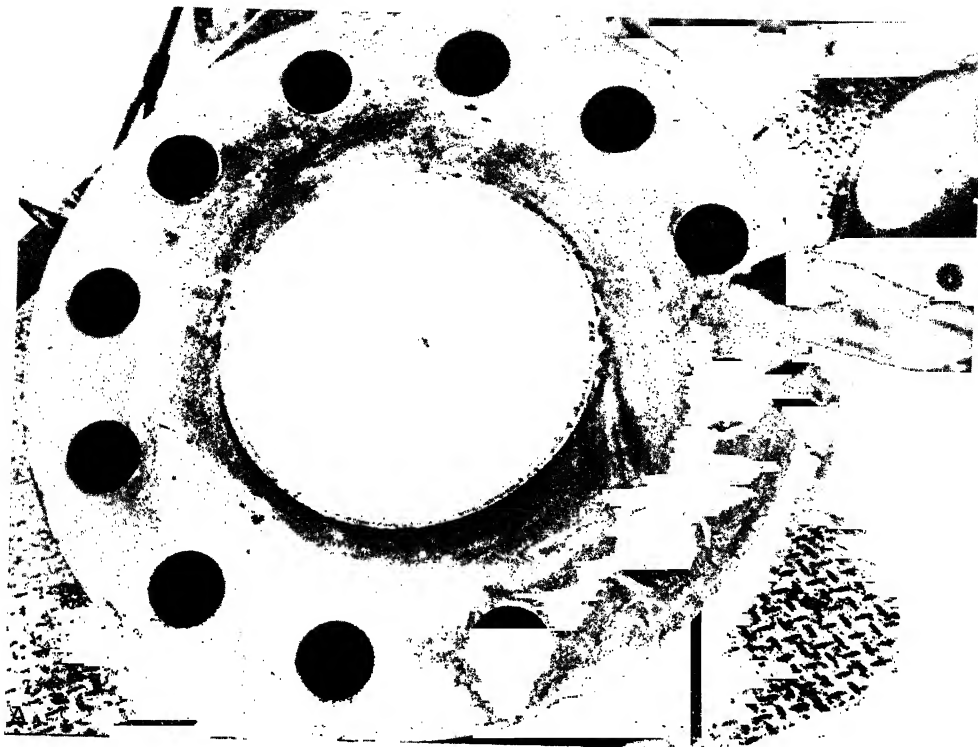
Principal Investigator - R.T. King

**OBJECTIVES** – This project is examining failed and used components from several pilot and bench-scale coal conversion plants now operating in the United States that provide the closest available simulation to service conditions anticipated in demonstration and commercial plants. Specifically, the goal is to accumulate materials and components durability and reliability data for the design and construction of pilot, demonstration, and commercial coal conversion plants; aid pilot plant and demonstration plant operators in providing timely failure analyses and recommendations on materials/component choices that will improve the operability of the plants; and provide materials performance data to a central DOE data bank for dissemination to other plant designers and operators.

**RECENT WORK AND ACCOMPLISHMENTS** – To discuss field experience with plant operators and to make the services of this program available, failure analysis teams visited several installations: Pittsburgh Energy Research Center; Synthane and Synthoil development facilities; Bureau of Mines Laboratory at Albany, Oregon; Bureau of Mines Laboratory at Bruceton, Pennsylvania; SRC facilities at Tacoma, Washington, and Wilsonville, Alabama; Westinghouse Gasifier at Waltz Mill, Pennsylvania; and Morgantown Energy Research Center. As a result of these contacts, the following failure analyses were performed: An analysis of tubes from a boiler that was operated for 1000 hr on No. 6 oil-coal slurry at PERC was completed, and data on the rate of formation of corrosion products were obtained that PERC interpreted as favorable to firing boilers in this mode. A type-310 stainless-steel heat exchanger tube that operated in a MERC fluidized-bed combustor in a  $760^{\circ}$  to  $927^{\circ}\text{C}$  ( $1400^{\circ}$  to  $1700^{\circ}\text{F}$ ) environment was examined. Relatively rapid attack at the fusion line of a longitudinal seam weld produced a notch varying in depth from approximately 8 to about 22 percent of the tube wall thickness. Particular attention should be paid to the behavior of seam welds if welded heat exchanger tubing is considered for later plants. Other tubing has recently been received for examination. Several failure analysis activities on components from SRC facilities were completed. The reports on failure analysis activities are on file with the Failure Analyses Center at the National Bureau of Standards.

**PLANS FOR THE COMING YEAR** – Failure analysis work will be continued on request. Examination of surveillance samples from liquefaction plants is expected to be part of the activity.





*(a) Type-347 Stainless Steel Flange Cover Plate after Several Years Service on Main Dissolver Tank SRC Plant;  
(b) Closeup Shows Cracking Identified near Welds Used To Attach Nuts to Plate*

## EVALUATION OF HEAT EXCHANGER MATERIALS

BATTELLE, COLUMBUS LABORATORIES

DOE - \$768,637  
5/28/76 - Continuing

**OBJECTIVES** – This program will obtain engineering data on the resistance of candidate heat-exchanger materials to corrosion/erosion in the bed of an atmospheric-pressure fluidized-bed coal combustor containing limestone as a sulfur sorbent, obtain engineering data on the resistance of candidate superheater tube materials to corrosion/erosion resistance of potential structural support materials operating uncooled in the bed, and investigate the performance of the low-temperature heat-exchanger tubes used to control the bed temperature of the fluid-bed combustor used in the research program. The materials under study for heat exchanger and superheater applications are: FSX 414 cobalt-base alloy; 310, 304, 347, and 18-18-2 stainless steels; IN 671 nickel-chromium alloy; P-9 chromium-molybdenum steel; and 304 coated with FeCrAl, and with Cr plus Al. Candidate support structure materials being studied are: 304, 310, and 347 stainless steels; Ni-Fe-Cr alloy 825; and pack aluminized 304 and 310 stainless steels. The alloy involved in the low-temperature heat-exchanger application is 316 stainless steel. All materials are being tested in the form of thin-wall turbine about 1-in. O.D.

**RECENT WORK AND ACCOMPLISHMENTS** – The program is divided into three parts: operation of the fluid-bed combustor facility, conduct of the corrosion/erosion research, and measurement of the velocity of the particulates in the bed. It is being conducted in a 24-in.-diameter atmospheric-pressure fluid-bed combustor using Illinois No. 6 coal and Greer limestone. Average bed temperature is 1620°F (992°C), superficial velocity is approximately 8 ft/sec, bed depth is 4 ft, excess air is about 25 percent, average Ca/S is 2.0, and the SO<sub>2</sub> content of the flue gas is approximately 550 ppm or 1.1 lb/MM Btu. The candidate heat-transfer materials have been placed on test for 10 hours, 500 hours, and 1500 hours at metal temperatures ranging from 1100° to 1580°F (593 to 860°C). The candidate structural support materials were exposed 1080 hours at 1620°F (882°C). The 316 stainless-steel low-temperature bed-cooling tubes were in service in the order of 1100 hours at a metal temperature generally not expected to exceed about 250°F (121°C). The results of the erosion/corrosion investigation indicate FSX 414 to be the best material for heat-transfer applications in the fluid-bed combustor, with 347 stainless steel being the runner up. Type 347 stainless steel could be expected to have a reasonable life when used in the freeboard of the combustor, while FSX 414 could probably provide an adequate service life in either the freeboard or the bed. Among the alloys being investigated as potential structural support materials, Incoloy alloy 825 is best with 347 stainless steel next. In the form of solid or heavy-wall components, these alloys (particularly, alloy 825) probably could be expected to give adequate service. Several of the 316 stainless-steel bed-cooling tubes eroded severely. They were at too low a temperature to form a substantial oxide scale; they formed only an oxide film. It appears evident that such a scale is necessary to protect the metal from erosion.

For the investigation of in-bed particle velocity two approaches were used. One was cinematography and the other was laser interferometry. The purpose was to gain information on velocity distribution and velocity/particle-size relationships to aid in understanding corrosion/erosion phenomena and to provide input for fluid-bed modeling studies. Individual particle-velocity measurements were made on more than 600 particles. The most probable velocity was found to be

0.5 m/sec while the average of all the data was 1.8 m/sec. About 30 percent of the data was in excess of the superficial gas velocity of 2.1 m/sec and 10 percent of the particles had velocities more than twice the superficial velocity. A few particles had velocities greater than 10 m/sec.

**PLANS FOR THE COMING YEAR** – The experimental runs have been completed and most of the work of evaluating the performance of the materials has been concluded. The major remaining item is the final report which is expected to be issued in December 1977.

## MATERIALS TECHNOLOGY FOR COAL CONVERSION PROCESSES

ARGONNE NATIONAL LABORATORY  
DOE - \$725,000  
7/75 - Continuing

**OBJECTIVES** – The objectives of this program are to evaluate refractories for slagging gasifiers, to develop and apply nondestructive evaluation methods for coal conversion systems, to predict corrosion reactions and their effects on the mechanical properties of candidate materials for demonstration plant components, to predict erosive wear rates of candidate materials under service conditions, and to determine the cause of failure of pilot plant components and recommend remedial action.

**RECENT WORK AND ACCOMPLISHMENTS** – Refractory corrosion tests using simulated Montana Rosebud slag have been conducted for 500 hours at 1500°C with oxygen partial pressures of  $\sim 10^{-3}$  Pa. Of the  $\sim 30$  refractories subjected to both basic and acidic slags under noncooled conditions, the best corrosion resistance ( $< 2$  mm loss) was exhibited by a fused-cast chrome-magnesia brick. When the apparatus was modified to provide prototypic thermal gradients by water cooling, silicon carbide refractories also performed well under high heat flux conditions (40 to 50 kw/m<sup>2</sup>). The silicon carbide has not performed well under the previous uncooled tests. Quantitative field measurements of the erosive wear in metal transfer lines have been carried out at the Bigas, Hygas, and Synthane pilot plants using ultrasonic techniques. For refractory-lined transfer lines, gamma radiography had been successfully used to obtain the erosive wear pattern. The feasibility of using acoustic emission to determine the optimum firing schedules for refractory linings has been established on a laboratory scale. An acoustic system for leaking valves detection has been developed and proven workable under field conditions. A survey of NDT needs in coal liquefaction was completed. Through the use of a computer program based on thermodynamic equilibrium to characterize the environments of various coal gasification processes, it was shown that the oxygen, sulfur, and carbon potentials varied widely among the processes even for conditions of identical pressure and temperature. An evaluation of corrosion-product phase stability for different commercial iron- and nickel-base alloys upon exposure to the multi-component gas environments indicated that sulfidation of structural materials is potentially the most severe process of material degradation in coal-conversion plants. The analysis also showed that, in general, iron-chromium-nickel alloys with high-chromium and low-nickel contents offer better resistance to corrosion by sulfur. Laboratory experiments on high-temperature corrosion of structural materials confirmed that oxygen-sulfur thermochemical diagrams can effectively describe material interactions in multicomponent gas environments. Experimentally determined growth rates for sulfide scales were  $10^3$  to  $10^4$  times greater than those for oxide layers. Also, high-nickel alloys exhibited severe corrosion from the formation of a liquid nickel sulfide. More than a dozen component failures were analyzed from the Synthane and Hygas pilot plants, the Grand Forks and Morgantown Energy

Research Centers, and the Battelle charburner. Reports and recommendations have been issued in all cases.

**PLANS FOR THE COMING YEAR** – Ongoing and future refractory/slag tests include the evaluation of the corrosion resistance of alumina and alumina-chrome based fused-cast brick and ramming mixes. These tests will all be done with prototypic thermal gradients. The nondestructive testing program will emphasize continued development of ultrasonic and infrared wear measurement systems, and acoustic emission for refractory firing control. A field usable prototype for valve leak detection will be completed. The effects of gaseous corrosion on the tensile properties of candidate materials will be evaluated after 1000-hr exposures. The kinetics of the multicomponent corrosion processes will be evaluated. The erosion testing rig will be used extensively in the development and verification of analytical models for the prediction of erosive wear under field conditions. Component performance and failure analyses will be performed as requested.

## HEAT EXCHANGER MATERIALS FOR FLUIDIZED-BED COAL COMBUSTORS

OAK RIDGE NATIONAL LABORATORY

DOE - \$105,000

6/1/76 - Continuing

Principal Investigators - R.H. Cooper, J.H. DeVan, T.G. Godfrey

**OBJECTIVES** – This work is evaluating the erosion/corrosion behavior of candidate heat-exchanger alloys in a coal-fired atmospheric fluidized-bed combustor (AFBC). Although crushed limestone or dolomite is employed in the fluidized bed as a sulfur absorber, the potential exists for both oxidation and sulfidation of the in-bed heat-exchanger tubes, which operate at wall temperatures from 500° to 870°C (930° to 1600°F). The durability and reliability of alloys for this application must be determined to make the AFBC viable for commercialization. The program is in direct support of the Technology Test Unit (supported under Coal Combustion and Cogeneration) in the Engineering Technology Division of ORNL.

**RECENT WORK AND ACCOMPLISHMENTS** – A small-scale AFBC was constructed by Fluidyne, and candidate heat-exchanger tube materials have been tested in the unit. The candidate materials included Incoloy 800, Inconel 600, types 304, 310, and 316 stainless steel, and aluminized alloy 800 and 310 stainless steel. These air-cooled tubes were exposed to the AFBC environment with weld temperatures ranging from 820° to 875°C, a Ca/S of 2.5 to 5.0, 2 to 3 percent excess O<sub>2</sub>, and a fluidizing velocity of 1.2 to 1.8 m/sec (4 to 6 ft/sec). A 500-hr surveillance test was completed; all tubing materials developed a tenacious and uniform CaSO<sub>4</sub> deposit 50 to 75µm (2 to 3 mils) thick with metal wastage less than 0.25 mm/year (10 mils/year), the lower limit of detection. Microprobe analysis revealed no evidence of sulfidation attack (hot corrosion). For 1000-hr additional exposure, 12 of the original and 4 replacement tubes of alloy 800, types 304 and 316 stainless steel, and Inconel 600 were inserted into the Fluidyne facility. The test was temporarily stopped because of failure of Inconel 600 tubes. The failed portions of the tubes were in areas of very low oxygen potential, which perhaps triggered a hot-corrosion response. All Inconel 600 tubes were removed from the experiment, and operation of the bed was changed to improve the air distribution. The 1000-hr test then proceeded without incident. The tubes were removed at the conclusion of the 1000-hr test (1500-hr total for some tubes). Although some of the tubes were bowed as a result of end restraint, their general condition was good with no evidence of severe pitting or rapid corrosion or erosion.

**PLANS FOR THE COMING YEAR** – Several of the 1500-hr tubes will be carefully examined by metallography, x-ray diffraction, and microprobe analysis. These tubes will be replaced and together with the remaining original tubes will be operated for an additional 3000 hr in the Fluidyne AFBC. Thus, corrosion/erosion data will be obtained for these candidate alloys for times to 4500 hr. In addition to these high-alloy tubes, tubes of 2 1/4 Cr-1 Mo/304 stainless steel will be included in the 3000-hr test for evaluation at temperatures in the range 480° to 590°C (900° to 1100°F), the temperature conditions of interest for utility applications.

## **HOT CORROSION/EROSION TESTING OF MATERIALS FOR FLUIDIZED-BED COMBUSTION**

EXXON RESEARCH AND ENGINEERING CO.  
DOE - \$871,124  
7/1/76 - 12/31/77

**OBJECTIVES** – The task is to operate the pressurized fluidized-bed coal combustor for 1100 hours to provide a test site and environment for exposure of specimens of heat exchanger alloys and gas turbine materials. These FBC exposures will provide corrosion/erosion data and comparisons of materials for application to advanced power systems using coal-derived fuels.

**RECENT WORK AND ACCOMPLISHMENTS** – Installation of the 21 heat-exchanger specimen probes (supplied by Westinghouse Research) and the turbine test section containing 24 blade specimens (supplied by General Electric) has been completed. All other PFBC of coal miniplant modifications were also accomplished in preparation for a shakedown test. The initial 100 hours of fireside corrosion materials exposure represents the shakedown phase of the program. Checkout tests have verified excellent temperature control of the heat-exchanger specimen probes. Difficulties have been experienced with the performance of the granular bed filter intended for final cleanup of fine particulates before entering the turbine test section. Flue gas temperatures greater than 1550°F have been maintained at the turbine section inlet with the aid of some methane injection.

**PLANS FOR THE COMING YEAR** – A 100-hr shakedown test will be conducted to assess the performance of the heat-exchanger material probes and to analyze the expected corrosion/erosion/deposition effects in the turbine test passage. It will be followed by the extended exposure tests of the heat-exchanger materials supplied by Westinghouse and the gas-turbine blade materials supplied by GE. Total exposure time will be up to 1000 hours (in 100-hour segments), with the tests interrupted between several segments to allow removal, inspection, and re-inspection of the specimens.

## **MATERIALS-PROCESS-PRODUCT MODEL OF COAL PROCESS TECHNOLOGY**

INTERNATIONAL RESEARCH AND TECHNOLOGY CORPORATION  
DOE - \$770,000  
6/1/75 - 12/31/77

**OBJECTIVES** – A model, based on the materials-process-product methodology (MPPM), is being developed for coal processing technology. The MPPM is an analytical tool in the form of an

an algorithmic structure that facilitates iterative, systematic evaluations of competing processes in response to exogenously specified variables, such as raw material specifications and costs, product specifications, process technology and costs (at the functional level of process breakdown), environmental limitations, tax policy, and financing costs.

**RECENT WORK AND ACCOMPLISHMENTS** – The MPPM has been functionally complete and available for use since June 1977. A draft final report was submitted to DOE in October 1977, which included a User's Manual. The model is installed on DOE's IBM 370 machine in Germantown, and is accessible from remote terminals under the Time-Sharing Option (TSO). The report contains typical uses of the model for policy analysis; for example, the effect on capital requirements and cost of production of substitute natural gas from coal are computed for variations in plant throughput, financing methods and costs, tax policy, coal sulfur content, raw-materials and byproduct prices, and changes in the process technology used. Environmental residuals are also evaluated parametrically by the model.

**PLANS FOR THE COMING YEAR** – The model data base will be extended to include more process technology options in the areas of gasification and control of environmental impacts. Model structure will be refined further.

## DESIGN AND EVALUATION OF COMPONENTS IN FOSSIL ENERGY SYSTEMS

### MECHANICAL TECHNOLOGY INCORPORATED

DOE - \$389,469  
8/15/77 - 8/14/79

**OBJECTIVES** – Engineering support services are being provided for the performance of selected tasks assigned by the DOE Components Branch encompassing the design and operation of machinery employed in coal conversion plants. The primary aim of the program is to aid field personnel in troubleshooting and improving machinery reliability for the purpose of minimizing downtime from mechanical equipment failures. The machinery performance histories accumulated under this program will also be useful to DOE organizations charged with the responsibility of selecting and maintaining equipment for future plants.

**RECENT WORK AND ACCOMPLISHMENTS** – A review and evaluation of failures, maintenance, and operability data on components used in the CO<sub>2</sub> Acceptor Pilot Plant, Rapid City, South Dakota, was conducted. A final report was written covering compressors, pumps, solids handling equipment, valves, and gas cleanup equipment.

**PLANS FOR THE COMING YEAR** – Task work assigned by DOE is expected to include field vibration measurements, analysis, and correction of existing mechanical equipment with chronic maintenance problems; analytical design audits; recommendations for improved specification writing; and design and testing of reliability improvements to existing mechanical components.

# STRUCTURAL MATERIALS TESTING FOR OXYGEN CENTRIFUGAL COMPRESSORS

SOUTHERN RESEARCH INSTITUTE

DOE - \$425,647

6/75 - 1/78

**OBJECTIVES** – This program is assessing the state-of-the-art of large centrifugal oxygen compressor construction and experimentally investigating the ignition and burn properties of materials that might be used to construct a 100-psi oxygen centrifugal compressor. The ignition temperature and burn characteristics of candidate rotor, stator, seal, diaphragm, diffuser, casing, and interstage cooler materials at various oxygen partial pressures, temperatures, and velocities are of interest.

**RECENT WORK AND ACCOMPLISHMENTS** – From discussions with manufacturers and users of centrifugal oxygen compressors, it was determined that several events may occur during operation that can lead to a compressor fire; therefore, it is desirable to use materials that are resistant to both ignition and propagation of a fire and to employ instrumentation to warn of unsafe operating conditions. Material evaluation test procedures in this program were designed to provide simulation of the oxygen conditions present in a compressor and the principal events that can lead to compressor fires. The oxygen conditions selected for the evaluation program included gas pressures of up to 1000 psi, gas temperatures of up to 500°F, and gas stream velocities of up to Mach 0.8. The high-velocity oxygen stream was directed toward the reduced gauge section of a torsion-type specimen by means of a flow nozzle. Frictional heating, which occurs when compressor components rub against each other, was simulated by resistively heating specimens at temperatures up to 1500°F by passing a low-voltage high-amperage current through the specimen. Unoxidized metal, which might appear in a compressor either during impact of foreign particles or removal of protective oxides by friction, was exposed to the oxygen by quickly applying a torque load to the specimen to fracture it at midgauge. These tests have been completed on 19 of 20 test materials. At 1000-psi oxygen pressure and Mach 0.8 oxygen velocity, aluminum has been found to ignite and burn at or below 500°F. Irons and carbon steels ignite at about 500° to 600°F and stainless steels ignite at about 700° to 900°F. Copper alloys, including bronze, tin bronze, and beryllium copper, ignite between 1000° to 1200°F. The nickel alloys evaluated ignite at about 1300° to 1500°F. The exact ignition and burn conditions are a function of the alloy, oxygen pressure, and oxygen velocity.

**PLANS FOR THE COMING YEAR** – The effect of foreign particle impact on ignition remains to be completed. A particle of glass will be shot at a velocity of about 1400 ft/sec against test specimens. The study should result in substantial information about the effects of particle impact, oxygen velocity, pressure, temperature, and localized alloy temperature on ignition and burning of metals in oxygen.

## VALVE TESTING AND DEVELOPMENT

MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$2,834,000

7/74 - Continuing

phases: evaluation of the capability of off-the-shelf valves to meet current lock hopper applications for pilot plant use during FY 1977 and 1978 and in the process to develop parametric valve design data and generate failure mode information for design improvements; and evaluation of prototype valve designs under simulated service conditions to compare the prototype design performance with off-the-shelf designs.

**RECENT WORK AND ACCOMPLISHMENTS** – Commercially available valves were surveyed, and 16 selected for purchase and testing. In addition, one no-cost cooperative agreement was signed with a manufacturer for testing their valve design, and several other agreements are pending. Provisions for testing valves up to 600°F have been added to both the static and dynamic test units. The reliability and versatility of these units was improved by modifying their instrumentation and control systems. A rationale for testing and statistical experiment design was completed for the dynamic unit. Support was provided to the MERC gas producer program to reduce the valve failures they were experiencing. This work included cooperation with the Albany Metallurgy Research Center of the Bureau of Mines and Argonne National Laboratory, for testing their valve leak-measuring equipment. A short-term study was conducted on gravity flow of solids. Results were encouraging and further work is being proposed in this area to size lines and valves more efficiently for solids handling.

Prototype valves for high-temperature solids handling service are being furnished under contracts awarded to Consolidated Controls Corporation and the Stratos Division of Fairchild Industries. The valves are to be delivered to MERC in the fall of 1978. The valves will be tested using a fluidized-bed unit for solids heating that will cover a temperature range from 350° to 2000°F. This unit will be designed and constructed by Combustion Power Company. A coal-water slurry system will likewise be used to test slurry lock hopper valves. This unit is in final design. Compressors were ordered for furnishing the high-pressure air and/or inert gas (2500 psig, 500 scf/m).

**PLANS FOR THE COMING YEAR** – A workshop is planned to bring together members of the valve manufacturing industry and Government and industry personnel working on development of components for coal conversion processes. Completion of the building and installation/shakedown of the automated data acquisition and process control equipment is planned. Complete testing of the valves will be completed during the year. Preliminary tests will be made on prototype valves using static test conditions (pressure, no solid flow, ambient temperature) and dynamics test conditions (valve design pressure, solids flow, ambient temperature). Final tests will be at design temperature and pressure using hot solids from the fluidized-bed heater.

## VALVE DEVELOPMENT FOR COAL GASIFICATION PLANTS

FAIRCHILD STRATOS DIVISION  
DOE - \$2,238,023  
6/3/76 - 6/2/80

**OBJECTIVES** – This program will develop a series of valves suitable for use in lock hoppers associated with high-Btu coal gasification systems. The valves will operate at 1600 psi at temperatures up to 2000°F with media consisting of gases, coal, dolomite, char, and slurry. Included in this effort will be pilot plant testing of eight 12-inch valves, and the design of valves sized for demonstration plants.



**RECENT WORK AND ACCOMPLISHMENTS** – Construction of a prototype valve has been completed for use as a test bed for developing components and seating elements. Feasibility of the design has been demonstrated with solids in the gas flow, and at elevated temperatures. Many combinations of sealing materials have been tested for abrasion and erosion resistance. Cycling of bearings has been performed at elevated temperatures with abrasive particles present. Sufficient testing has been performed to justify proceeding with the design of the initial quantity of deliverable valves.

**PLANS FOR THE COMING YEAR** – After approval of the Phase I components testing effort, design will proceed for the deliverable 8-inch Phase II valves. These will be fabricated and initially tested at Fairchild Stratos. They will then be forwarded to Morgantown, West Virginia (where a test facility is currently under construction) for testing with simulated coal gasification conditions. Using the information gained from this program, the pilot plant size valves (12 inch) will be designed, fabricated and tested during Phase III (1979-1980) of the current program.

## **DEVELOPMENT OF WEAR RESISTANT VALVE MATERIALS**

**U.S. DEPARTMENT OF THE INTERIOR, BUREAU OF MINES**

**ALBANY METALLURGY RESEARCH CENTER**

**DOE - \$975,000**

**2/75 - Continuing**

**OBJECTIVES** – This project will develop, test, and evaluate materials that have abrasion, adhesion, and erosion resistance to operate in critical valves in coal gasification systems. A further objective is to manufacture useful valve hardware for trial insertion in operating gasifiers. The need for improved materials has been demonstrated by frequent gasifier valve failures, some of which have caused premature gasifier shutdown. For example, a butterfly valve in a product gas line of the MORGAS plant failed by erosion in less than 40 hours. Ball valves in the same plant have leaked excessively through the wear scars caused by adhesion and abrasion of sealing surfaces. Char slurry letdown valves in the Synthane plant have failed by erosion. The wasteful and bulky slurry system itself is necessitated only because no valves exist to remove char at 1000°C. Could such a system be designed, the energy which is presently lost by cooling and reheating the char could be saved, and the char could be burned to produce steam as it leaves the gasifier.

**RECENT WORK AND ACCOMPLISHMENTS** – The Albany program is geared to generate wear data that will be useful to design engineers. Work to date has consisted of (1) laboratory testing materials for wear resistance, (2) simulation testing of materials as parts in full-scale ball valves, and (3) producing valve parts from promising materials for trial in coal gasifiers. An elevated temperature erosion testing machine was built in FY 1976. Samples in the machine are blasted with a gas-propelled stream of fine alumina powder to simulate an erosive gasifier stream or a high velocity leakage through a valve. Over 3000 screening tests have been run at elevated and room temperature. The tests have shown that most metals, regardless of their prior history, erode similarly. However, dense ceramics such as WC, SiC, B<sub>4</sub>C, Si<sub>3</sub>N<sub>4</sub>, TiC-Al<sub>2</sub>O<sub>3</sub>, and TiB<sub>2</sub> are highly erosion resistant. Coatings of these materials when applied by CVD, sputtering, or electrodeposition in sufficient thicknesses also have been found to be highly erosion resistant. Other erosion resistant materials include WC and Mo that have been boronized. Materials selections for valves, nozzles, and pumps have been made as a result of the Albany erosion data bank. A Falex No. 6 thrust washer wear testing machine is used to approximate the wear encountered in valves when two mating surfaces rub against one another. Several superior materials have been identified.

A lock hopper simulator was designed to evaluate materials as components in a 6-inch ball valve. The valve body is rotated 180 degrees around the ball which is anchored to keep it motionless. With each rotation, char or ash is transferred through the valve to represent one lock hopper open-close cycle. Wear is monitored by periodically performing a leakage test across the ball. Commercial sources have donated valves for testing. Valve parts that were cast and finished at Albany have been supplied to the MORGAS and Synthane pilot plants for in-service testing. Five 10- and 12-inch ball valve seats logged over 2000 hours of operation in coal-feed and main ash discharge lock hopper valves, and six 25 percent Cr white iron 6-inch seats have successfully replaced teflon seats in cyclone ash-discharge lock-hopper valves. Three butterfly valves were tried unsuccessfully in the MORGAS plant. A fourth valve with a WC liner and a tool steel bonded TiC disk is awaiting trial. In the Synthane plant, alumina and boronized Mo choke valve liners have performed successfully in the char slurry letdown system. Additionally, liaison has been maintained with other laboratories, interested valve manufacturers, and conversion plant personnel.

**PLANS FOR THE COMING YEAR** – Erosion and other wear-related screening tests will be continued, but a greater emphasis will be placed upon delineating the properties of promising materials and upon exploring coating techniques such as pack cementation, diffusion, and high rate sputtering. More parts will be made for trial in operating gasifiers in an effort to correlate field experience with laboratory data and to demonstrate that certain manufacturing processes will work. Materials will include D-gun coatings, white iron, TiB<sub>2</sub>, and boronized metals. Lock hopper simulator tests at Albany will continue.

## **TURBINE MATERIALS EVALUATION IN COAL-FIRED FLUIDIZED-BED COMBUSTION**

GENERAL ELECTRIC COMPANY  
DOE - \$240,000  
7/1/76 - 6/30/78

**OBJECTIVES** – This program is providing 1000-hr engineering data on the corrosion/erosion deterioration of gas-turbine materials exposed to the exhaust gas from a pressurized fluidized-bed combustor. Both laboratory exposure under this program and in-plant exposure under a separate contract will be used to gather these data. On this basis, an estimate of parts lives will be made for several assumed contaminant levels and operating temperatures and pressures.

**RECENT WORK AND ACCOMPLISHMENTS** – Tasks that have been completed include preparation of a work plan, alloy selection for corrosion tests, establishment of test conditions for the corrosion tests, and design and construction of a turbine test section and airfoil shaped specimens for insertion into cascades in the test section. The test section with test specimens in place was shipped to Exxon-ERE and installed in the Miniplant (PFBC). A 100-hr shakedown test of the turbine test section in the Exxon Miniplant was begun in late November 1977 and stopped after 17 hours because of a high pressure drop across the granular bed filter. Indications were that the turbine test section performed satisfactorily to this date. The specimens were inspected and showed no evidence of corrosion or erosion although there were deposits. They were removed and are being analyzed. The shakedown test was continued for the balance of the 100 hours. There was some problem with plugging of a pressure reduction station upstream of the cascades during the last part of the shakedown test. Corrosion testing of the selected alloys in the small-burner rig has accumulated about 2000 hours under a laboratory simulation of the PFBC environment. Some problems

have arisen during the course of the 2000-hr small burner rig tests with regard to the environmental simulation. These problems are presently being resolved.

**PLANS FOR THE COMING YEAR** — Small-burner rig tests will continue for up to 7000 hours of testing. A 1000-hour materials test will be initiated and completed in the Exxon Miniplant. Test specimens from the small-burner rig tests and the 1000-hr Miniplant test will be evaluated. Corrosion data from these tests will be interpreted, and the final report will be prepared.

## **DEVELOPMENT OF PARTICULATE CONTROL CYCLOCENTRIFUGE**

**MECHANICAL TECHNOLOGY INCORPORATED**

DOE - \$485,270

5/76 - 8/78

Principal Investigator - J.T. McCabe

**OBJECTIVES** — The efficiency of a mechanical aerodynamic device called a Cyclocentrifuge will be demonstrated for use in controlled separation of fine particulate matter from a hot, pressurized gas stream. The objective of the present phase is to demonstrate the separation principle in a cold-flow laboratory test loop. High efficiency cleanup of hot, pressurized gas in a continuous and reliable manner is essential to fossil energy utilization in a number of areas, one of which is the economic use of low-Btu gas. Careful particulate control must be exercised when low-Btu gas is used as fuel gas for either the retrofitting of a natural gas fired steam generating unit or for feeding the combustor of a gas turbine. Another application area, equally as important as the final cleanup of gas, is limiting the particulate loading to large capacity liquid scrubbing systems where the quantity of effluent must be minimized.

**RECENT WORK AND ACCOMPLISHMENTS** — Design studies have been completed and a laboratory-scale Cyclocentrifuge handling 1100 scf/m of gas is currently being manufactured. This size was selected to facilitate testing accuracy and also to accommodate a specific hot gas test facility without scaling the laboratory model. A dust injection system and a particulate measuring scheme have been devised to provide accurate test results in the particle size range from ½ to 10 microns. Calculation of the theoretical grade efficiency curve predicts that the 50 percent efficiency point will occur at about ½ micron.

**PLANS FOR THE COMING YEAR** — Laboratory evaluation of the separation principle will be completed. Further plans include test evaluation using hot pressured producer gas. Further laboratory studies of design parameters are also planned.

## **DEVELOPMENT OF A STEAM-DRIED COAL SLURRY FEED SYSTEM**

**MORGANTOWN ENERGY RESEARCH CENTER**

DOE - \$520,000

6/76 - Continuing

**OBJECTIVES** — A steam-dried coal slurry feed system is to be developed and tested as a means of injecting dry, crushed coal into a high-pressure process. Such a system could eliminate the need for troublesome high-pressure valve systems on coal feed lines.

**RECENT WORK AND ACCOMPLISHMENTS** — System design was completed, a site was selected, the structure was erected, and installation is now approximately 85 percent complete. A cold-test system was assembled for studying the effectiveness of various nozzles, and West Virginia University was contracted to design steam-slurry nozzles for the MERC dryer. Planned completion date for the dryer system is scheduled for December 1977. A nozzle testing facility was assembled in the MERC pilot plant. The design of an initial nozzle was completed by West Virginia University and MERC personnel, and the nozzle was fabricated. Preliminary testing of the nozzle in the new facility was successful, and further tests are underway. Pneumatic nozzles or, more properly, two-fluid nozzles, appear to be best suited for the vertical lift dryer application. Such nozzles achieve atomization by internal or external mixing of the liquid or slurry with a second pressurized fluid. External mixing eliminates the erosion and plugging problems anticipated with single-fluid nozzles; therefore, initial operation of the process will incorporate external mixing via a two-fluid nozzle concept. A vertical lift dryer simulation study made by C.F. Braun and Co., was reviewed and the computer program used in this study will be adapted for use in predicting operating parameters of the MERC system. Several important conclusions were reached supporting the planned MERC studies:

- Vertical lift dryers are feasible if coal particles can be kept from coalescing on the dryer walls; thus MERC will determine coalescing tendencies experimentally.
- The spray size and flow pattern are the most important parameters in determining the performance of a dryer; the effect of spray flow pattern on dryer length must be experimentally ascertained.
- Spray droplet size should be at least as fine as  $300\mu$  and the spray should not impinge on the dryer wall until the gas has cooled to  $700^{\circ}\text{F}$  or less, thus MERC cold study tests are planned to study these parameters.
- Required residence times in the dryer are on the order of 0.7 to 1.5 seconds; MERC calculations yield values from 0.2 to 1.3 seconds for  $500\mu$  droplets (maximum residence time of the MERC system at full capacity is 4.3 seconds, which should be ample).
- Temperature difference of  $50^{\circ}$  to  $80^{\circ}\text{F}$  between the saturation temperature and the dryer effluent temperature is a sufficient driving force to dry the coal; this temperature difference will be approximately  $199^{\circ}\text{F}$  for the 250 psi MERC test system and at least  $55^{\circ}\text{F}$  for a 1000 psi commercial system.
- The Braun study assumes use of pretreated coal to minimize agglomeration; the MERC program will not utilize coal pretreatment, and consequently the tendency for coal agglomeration in the dryer bottom will be studied.

**PLANS FOR THE COMING YEAR** — After completion of system construction and instrumentation, a shakedown for unit operation will be made followed by a test using anthracite coal in a 50 percent water slurry with coal ground to 70 percent through 200 mesh. Testing of similarly sized bituminous coals will be started before the decision concerning the use of recycle steam is made.

## INDUSTRIAL COAL CONVERSION EQUIPMENT CAPABILITIES

OAK RIDGE NATIONAL LABORATORY

DOE - \$300,000

10/1/76 - Continuing

**OBJECTIVES** – Surveys will be conducted of industrial equipment capabilities that will identify the present ability of industry to supply the equipment needed for coal conversion demonstration plants. The project will also determine research and development (R&D) needs, including lead-time requirements, for producing advanced design equipment for the various unit operations that are of critical importance to DOE coal conversion programs.

**RECENT WORK AND ACCOMPLISHMENTS** – Four subjects were covered in the surveys initiated in FY 1977: rotating components, valves, hot-gas cleanup devices, and heat recovery equipment. The first subject covers rotating machinery required in one or more coal conversion processes. The equipment includes centrifugal and axial compressors, liquid and slurry pumps, drives such as motors and turbines, and energy recovery devices such as expanders. Lists of the four types of equipment requirements were developed from information on several processes including Synthoil, Lurgi, Coalcon, Fischer-Tropsch, Hydrocarbonization, HYGAS, and Bi-Gas. A letter was prepared for the *Commerce Business Daily* to obtain an indication of industry readiness to supply information on their present capabilities for design and manufacture of rotating equipment for coal conversion facilities, as well as to comment on R&D needs. Questionnaires concerning all of the equipment were sent to the appropriate industrial firms, and an analysis was made in the form of draft reports on the four subjects. It was concluded that rotating equipment should be available for nearly all expected clean-stream applications likely to be found in coal conversion facilities with the exception of oxygen compressors. Manufacturing facilities are available for valves; however, there is a lack of design and operating experience for the severe service conditions expected for the critical valves. At this time, the use of high-temperature, high-pressure (HTHP) filtration in gasification applications appears to be limited due to the technical difficulties associated with its realization and the marginal economic advantages accruing from its use. In terms of equipment: (1) Inertial separation devices have most likely achieved their maximum levels of efficiency with existing designs, and research aimed at dramatically improving their performance appears to be unwarranted. (2) Surface filters of either the Brunsmet metal type or unique high-temperature fabrics have potential in HTHP gas cleaning applications. This choice is subject to the constraint that sufficiently high throughputs coupled with acceptable operational life be demonstrated. (3) Granular bed filters, particularly of the more advanced designs (Ducon and Combustion Power Company), are at the stage where on-line operational testing is needed to ascertain their overall filtration characteristics. (4) Major engineering and operational problems need to be resolved before electrostatic precipitators become viable HTHP filtration devices. (5) Commercial equipment for the removal of  $H_2S$  at high temperatures is unavailable at this time. In fact, the most developed system, Morgantown Energy Research Center's fixed bed iron oxide process, is still in the pilot plant stage. From an environmental point of view, any HTHP filtration system capable of satisfying turbine particulate allowances will more than likely be able to meet any EPA emissions constraints. About two-thirds of the firms contacted responded to the heat recovery questionnaire. Most indicated that the design of equipment was more difficult than the fabrication. There is a need for physical property information on the coal conversion materials. The high solids content of some streams is a problem. Where both high temperature and high pressure are involved, there may be a problem in heat exchanger design.

**PLANS FOR THE COMING YEAR** – The survey project will be continued through FY 1978. Final reports on the four surveys completed in FY 1977 will be issued. The surveys will be expanded to include consideration of operating experience and reliability of equipment. Cost and required lead time for available equipment will be determined. Development costs for equipment not now available will be included.

## COAL EQUIPMENT TEST PROGRAM

OAK RIDGE NATIONAL LABORATORY  
DOE - \$280,000  
7/1/76 - Continuing

**OBJECTIVES** – This program will examine specific equipment requirements for coal conversion demonstration plants being designed under DOE-industry contracts. Components which can be considered critical to successful performance of these plants will be developed as well as cost effective means to evaluate the performance of these components prior to plant startup. The program will identify potentially troublesome components and, thus, decrease startup problems and prevent unscheduled shutdown of the plant.

**RECENT WORK AND ACCOMPLISHMENTS** – A study was completed in cooperation with Stearns-Rodger Engineering Company, Denver, Colorado, of the facility requirements for testing demo plant-scale valves, slurry pumps, gas cleanup systems, and solids feeders. The results indicate such tests were feasible and useful, but costly. Currently, ORNL, assisted by TRW Energy Systems, McLean, Virginia, is initiating work with four demonstration plant subcontractors: Illinois Coal Gasification Group, Cogas Process; Conoco, Slagging Lurgi Process; Memphis Light Gas and Water, U-Gas Process; and W.R. Grace, Texaco Coal Gasification Process. Work performed by these groups will identify potentially critical components and develop plans for testing these various pieces of equipment in available or modified facilities at energy research centers, national laboratories, pilot plants, or other appropriate locations. In parallel with the above activity, ORNL is working with DOE subcontractors who are developing advanced coal feeding equipment: Ingersol-Rand Research, Inc., Princeton, N.J.; Foster-Miller Associates, Inc., Waltham, Massachusetts; and Lockheed Missiles and Space Co., Inc., Sunnyvale, California, to make appropriate plans for testing pilot plant-scale (~5 t/hr) feeding systems when they become available.

**PLANS FOR THE COMING YEAR** – In cooperation with our subcontractor, TRW Energy Systems, efforts will continue to define demonstration plant critical components. System flow diagrams, operating conditions, and equipment lists will be examined as they become available from demonstration plant subcontractors. Potentially critical components will be identified and further examined to establish priority for the most urgent few which can be effectively tested. Test sites will be surveyed and test programs defined. Following the choice of test sites, subcontracts will be issued for the development of preliminary designs, schedules, and cost estimates for facility modifications and test program execution. In parallel with the above activities, ORNL will work with personnel from the Jet Propulsion Laboratory, Pasadena, California, who are coordinating the DOE advanced solids feeder development program, to develop test sites for this equipment when prototypes become available.

## LOCK HOPPER VALVE DEVELOPMENT

### CONSOLIDATED CONTROLS

DOE - \$2,547,000

5/28/76 - 5/27/80

**OBJECTIVES** – This project involves the design, manufacture, test, and evaluation of 8- and 12-in. valves that would be compatible with operating conditions found in numerous coal gasification plants. Such valves will be required to assure the efficient, continuous, and safe operation of coal conversion processes in future plants. Specific objectives are three-fold: provide reliable data sufficient to permit the confident design of coal gas valves compatible with existing and projected pilot plant operations; produce reliable coal valves suitable for commercialization; and provide for the orderly transfer of the demonstrated technology to private industry.

**RECENT WORK AND ACCOMPLISHMENTS** – The valve chosen for preliminary design studies and prototype development will incorporate high alumina ceramic and commercial refractory parts for higher reliability and longer life because of the higher resistance to corrosion and erosion over metal parts. An extensive material survey and evaluation was conducted to select a refractory material that in addition to its insulating qualities, would retain a high degree of structural integrity as a finished component when used at elevated temperatures and pressures under the highly erosive and corrosive environments. High alumina ceramic components are used in areas where strength greater than that offered by any refractory material is required. These components include such items as the seats, the sealing disc in the gate, stop rings, and—most noticeably—the ceramic spring. All components have evolved from the preliminary design conception to final configuration with little design change because of the long manufacturing time by the ceramic industry. With all material and components on hand, assembly of the first prototype valve is about 50 percent complete. A test fixture was fabricated to allow gaseous nitrogen under pressure to permeate a cylindrical sample. Data from the Plicast 40 and Taycor 414 FH samples indicated the Plicast has about three times the permeability of the Taycor material at 1600 psig. The Greencast samples showed the least amount of permeability. These materials have also been subjected to shear tests, linear expansion tests, and fabrication of structural gates. Other detailed test procedures have been established.

**PLANS FOR THE COMING YEAR** – Prototype valves (8-in.) will be fabricated and tested. The test results will be analyzed to identify necessary modifications. Based on these results, 12-in. valves will be fabricated and tested.

## ALLOY EVALUATION - LIQUEFACTION

### AMES LABORATORY

DOE - \$90,000

10/1/76 - Continuing

Principal Investigator - T.E. Scott

**OBJECTIVES** – Commercial coal liquefaction dissolvers will probably be constructed of 2 1/4 Cr-1 Mo steel with a weld overlay of stabilized austenitic stainless steel on the inner wall. Clearly, the stainless-steel inner lining should serve to resist, or at least mitigate, the aggressive environment of the dissolver. In the probable event of localized loss of the stainless-steel liner during operation, the 2 1/4 Cr-1 Mo steel will be exposed to coal slurry, high-pressure hydrogen and dissolver reaction

products. Accordingly, the initial objective of this project is to evaluate the effect of a coal liquefaction dissolver environment on the mechanical property integrity of 2 1/4 Cr-1 Mo steel.

**RECENT WORK AND ACCOMPLISHMENTS** — Baseline mechanical properties of notched and smooth bar tensile samples of 2 1/4 Cr-1 Mo steel have been determined subsequent to their exposure for 168 hr under the following conditions: 2000 psig argon gas at 500° and 900°F, 4000 psig argon gas at 500° and 900°F, 2000 psig argon gas at 500° and 900°F, while being stressed at 48,600 psi. These samples were all tensile tested under ambient conditions after the exposures cited above (120 tests). Other samples in the as-received condition were tensile tested at 72°F in air or at 500°F or 900°F in argon at atmospheric pressure making a total of 180 tests. Results of these tests revealed that no deterioration of the mechanical properties of 2 1/4 Cr-1 Mo steel occurred during these exposures.

**PLANS FOR THE COMING YEAR** — A nearly-completed facility designed for safe operation with high-pressure hydrogen will house recently acquired pressure vessels designed to accommodate coal slurry and hydrogen at pressures up to 5000 psig. After installation and checkout of the pressure vessels, notched and smooth bar samples of 2 1/4 Cr-1 Mo steel will be exposed for 168 hr with and without applied stress, in a coal slurry at temperatures of 500° and 900°F and hydrogen pressures of 2000 and 4000 psig. Subsequent to the hydrogen-coal slurry exposures, the samples will be tested under ambient conditions and the results will be compared to the baseline data (described in the preceding paragraph) to determine whether the simulated dissolver environment adversely affects the properties of 2 1/4 Cr-1 Mo steel.

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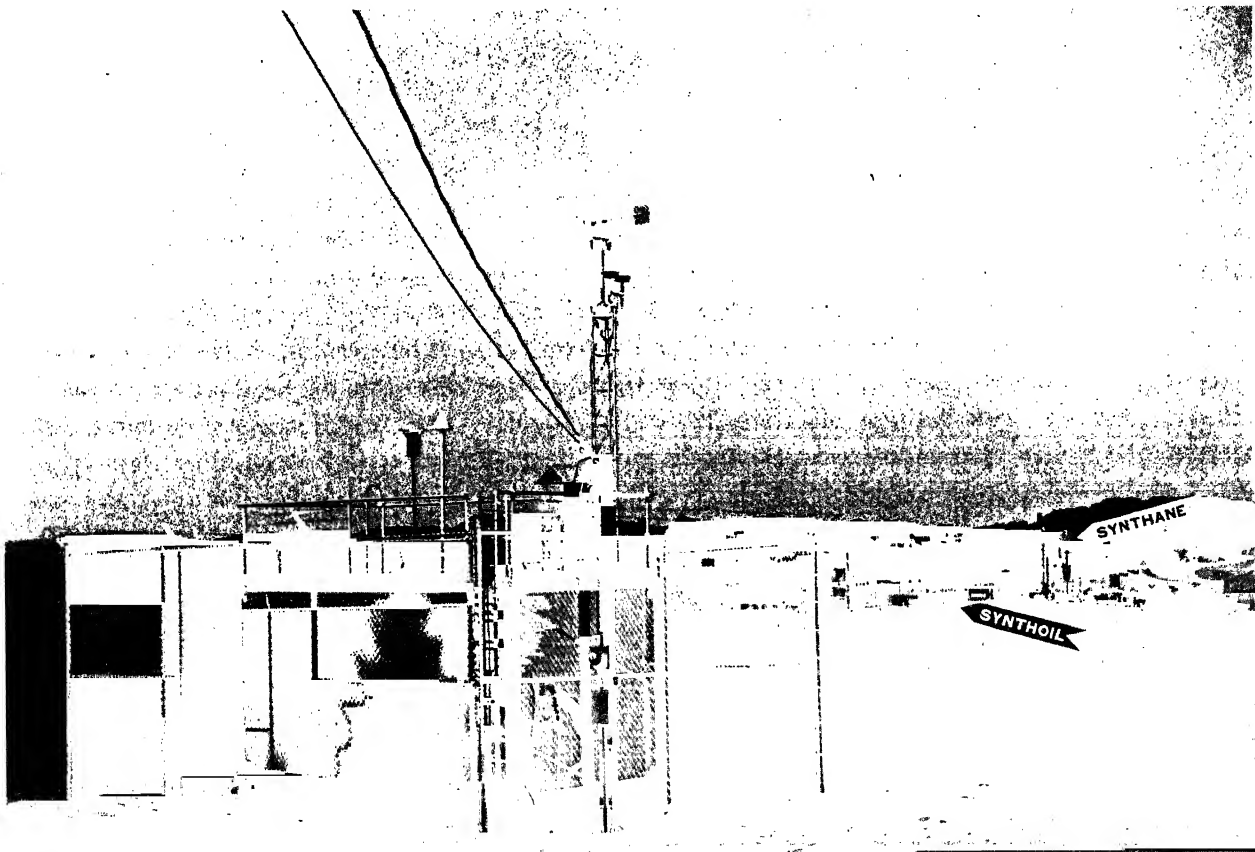
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## ***PROGRAM CONTROL AND SUPPORT***

Program Control and Support (PCS) activities provide specialized support for the DOE Fossil Energy technology programs. These activities cut across all the project-oriented endeavors of the Fossil Energy divisions. Thus, PCS serves an intermediary role by coordinating the programmatic interests of the Divisions with the broader national perspective of the Fossil Energy Program Director. PCS provides centralized intermediate and long-range FE-related planning; consolidation and preparation of the FE budget; an independent evaluation of project and program accomplishments; expertise in process engineering and economics; environmental analysis; and a variety of administrative services for the Divisions and the Program Director. Activities also include economic and systems studies of various FE efforts or proposed efforts. Environmental assessments and coordination with Government and industry environmental interests are centralized in this area, as are energy conservation efforts within FE activities.

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*View of Continuous Ambient Air Monitoring Sited Trailer; Site 5 View to West Overlooking Synthane and Synthoil Pilot Plants*

## PROCESS AND PROGRAM ANALYSIS RESEARCH STUDIES

OAK RIDGE NATIONAL LABORATORY

DOE - \$1,350,000

8/1/76 - Continuing

**OBJECTIVES** – These studies provide technical and economic evaluations of competing processes and systems for coal conversion and utilization. The subprograms involved are low-Btu gas, high-Btu gas, direct combustion, liquefaction, and advanced power/combined cycle. Also included are general subjects such as beneficiation, gas cleanup, transportation, and the potential for synthesis of chemicals from coal.

**RECENT WORK AND ACCOMPLISHMENTS** – The low-Btu gas studies included technical evaluations of 99 processes that produce low- and intermediate-Btu (150 to 500 Btu/scf) fuel gas from coal; 21 were selected for more detailed study. Descriptions were prepared, and the processes were compared generically and specifically. The direct combustion effort was limited to a study of conversion to coal in the industrial sector. This study identified the important factors that restrict the wide acceptance of direct coal combustion in industry, and explored some possible approaches to overcoming these barriers. Environmental and financial barriers were considered, as well as economic comparison with the alternatives. The economic analysis explored the effect that size, coal quality, and region of the country have on the incentives for an industrial steam user to convert to a coal combustion system. The proposed Federal fuel taxes and investment tax credits were also examined to determine their effect on the decision. The technologies considered included conventional boilers with and without scrubbers, fluidized-bed combustion, and the firing of a coal-oil mixture. Results indicate that at least for the near term, extensive conversion from oil and gas to coal combustion does not appear to be forthcoming. The lack of substantial economic incentives, increased risk because of intensified capital requirements, and the lack of a consistent environmental policy lead industry to postpone any energy decisions. Industrial boiler fuel taxes and tax credits will motivate some of the large steam users to convert; however, for the small and medium users, the incentives are lacking and risks appear to be too high. One part of the study examined the relative advantages and disadvantages of the various advanced power conversion systems being considered by DOE-FE. Emphasis was on technological aspects of the concepts, especially experimental experience in the development of the critical components essential to the technical or economic feasibility of the concept. Background performance information was compiled, rate of development of key components (such as compressors and turbines over the past several decades) determined, and, where possible, R&D expenditures on key components summarized. This information was gathered and drafted into a set of 12 topical reports: steam power plants for central stations; open-cycle gas turbines; open-cycle gas turbine-steam combined cycles; open-cycle coal-fired gas turbines; air and water cooling of gas turbine blades; ceramic gas turbines; closed-cycle gas turbines; supercritical CO<sub>2</sub> cycle; alkali metal-vapor cycles; open-cycle MHD systems; fuel cells; and comparison of advanced energy conversion systems on the basis of R&D experience. Other studies include liquefaction processes under subcontract by the Ralph M. Parsons Company; high-Btu gasification under subcontract by the Scientific Design Company; and determining the potential for the synthesis of chemicals from coal under subcontract by the Radian Corporation.

**PLANS FOR THE COMING YEAR** – Reports on the above work will be completed and issued. The work will be continued with emphasis on specific processes of interest to DOE. Subjects being

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considered include: beneficiation versus product gas cleanup, computer modeling of the HYGAS gasifier, and in situ gasification for the production of gasoline via the methanol route.

## ESCOE ENGINEERING PROGRAM

### ENGINEERING SOCIETIES COMMISSION ON ENERGY

DOE - \$3,067,892

11/76 - 11/79

**OBJECTIVES** – The ESCOE Engineering Program was established, in response to a request from the U.S. Energy Research and Development Administration, by the five founder engineering societies: American Institute of Chemical Engineers; American Institute of Mining, Metallurgical, and Petroleum Engineers; American Society of Civil Engineers; American Society of Mechanical Engineers; and Institute of Electrical and Electronics Engineers. This program's purpose is to conduct independent technical and economic evaluations in the areas of energy production, conversion, and use, and to advise DOE in these areas. These evaluations are used in the planning and management of energy RD&D activities within the Federal Government. An additional objective is the transfer of relevant energy information to and from the technical community. The professional staff is to consist of the Executive Manager and a team of about 10 Engineers-in-Residence. Each Engineer-in-Residence will have at least 8 years of relevant energy experience and will be on loan from his sponsoring employer for about 2 years. Engineers-in-Residence will come from various energy supply, technology, and service companies and universities. Each engineer is fully paid during his residency by ESCOE and is expected to return to his sponsoring employer at the completion of his residency.

**RECENT WORK AND ACCOMPLISHMENTS** – The ESCOE Engineering Program was initiated early in FY 1977. The major effort in this fiscal year has been the establishment of the program in Washington and the staffing of the ESCOE professional team. The Executive Manager was named and commenced his assignment on April 15, 1977. Two Engineers-in-Residence have been appointed and three additional candidates have accepted appointment to positions beginning in October 1977. More than 200 applications were screened by the Engineers-in-Residence Selection Committee. A continuing effort is underway to solicit additional applications and nominations in specific areas of expertise. The first three technical tasks were defined and scheduled to begin on October 3, 1977:

- *Coal Conversion Comparisons.* ESCOE will appraise many alternative processes for the production of liquid and gaseous fuels from coal and recommend the R&D areas that should be most productive to provide viable commercial clean fuel from coal processes. At least seven processes for the conversion of coal into liquid or gas fuel are presently receiving a significant amount of Government support for R&D. Each process is being pursued by a different group. ESCOE will make a complete identification of process conditions for each and study the fundamental economics. Capital and operating costs will be examined and restated on a consistent basis.
- *Technical Research Information.* ESCOE will develop a concise fossil energy information system that will be used in carrying out all tasks in support of the DOE Fossil Energy Program. The first element of this task includes the definition of the information base

and information sources, selection of information retrieval methods, and the development of technical documentation standards.

- *Examination of Public Nonfossil Energy Research for Potential Applications to DOE Fossil Energy Technology Development.* The accelerated pace of energy-related R&D in the United States has made it difficult for individual researchers and program managers to maintain a continuing awareness of relevant research outside their immediate areas of interest. In support of the DOE Fossil Energy Program, ESCOE will examine major research programs in areas such as solar, geothermal, and nuclear technology to determine possible utilization of research and technology by the fossil energy program and to identify research efforts that might best be done under the joint auspices of fossil and nonfossil R&D programs.

**PLANS FOR THE COMING YEAR** – Individual tasks will be developed as a function of DOE needs and ESCOE's perspective of where ESCOE efforts can best assist in resolving national energy problems. Specific task assignments will be developed between relevant DOE and ESCOE staff under the supervision of and final approval by the DOE Technical Representative and the ESCOE Executive Manager. To assist in development of new tasks and coordination of R&D efforts between major Government and industry energy technology programs, a Task Review Committee will be established consisting of senior energy technology policy staff from the Electric Power Research Institute, Gas Research Institute, National Coal Association, DOE, ESCOE, and other Government and industry groups. As a part of the ESCOE Engineering Program responsibility to communicate relevant energy technology matters to working engineers and as a part of the continuing recruitment effort, a newsletter—the ESCOE Echo—will be issued beginning November 1, 1977.

## **NATIONAL ENERGY POLICY PROJECT**

**THAYER SCHOOL OF ENGINEERING, DARTMOUTH COLLEGE**

**DOE - \$449,780**

**11/75 - 1/13/78**

**OBJECTIVES** – The technological, environmental, and economic implications of potential energy supply/demand scenarios are needed by U.S. policy designers. This project is developing a set of long-range planning tools and analyses to enhance the decision making process by projecting the impact on the U.S. energy system of variables such as different rates of R&D, earlier commercial availability of advanced energy technologies, and other possible policies, regulations, and international events.

**RECENT WORK AND ACCOMPLISHMENTS** – The focus has been on the development of a major, disaggregated computer model of U.S. domestic energy supply. Studies completed in 1977 using FOSSIL 1 included an evaluation of the proposed National Energy Plan and an analysis of coal industry trends for the EPRI-sponsored Energy Modeling Forum. The U.S. energy system is simulated over the years 1950 to 2020, using an interactive set of differential equations. Energy supply is modeled through 23 production types: four for coal, five each for oil and gas, eight for electricity, and one for solar-geothermal heating. End-use demands are a function of energy price and availability and an exogenously-supplied GNP. Secondary demands for primary fuels by elec-

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tricity and synfuels are explicitly included. OPEC imports are also internally modeled, with strong supply/demand feedbacks to domestic oil production. FOSSIL 1 results indicate the following trends for the next several decades:

- Coal acts as a swing fuel, filling gaps in oil and gas consumption in all areas; it is used increasingly by utilities, as a feedstock for synfuels, and by commercial and private consumers
- Nuclear utilities, along with coal-fired utilities, produce the largest share of electricity generation; oil- and gas-fired plants are delegated to peak-loading and thus play a continuously smaller role in total generation
- To sustain the current oil-based economy, the United States continues to increase its dependence on OPEC nations as suppliers of oil—a trend that continues until an oil synthetics industry becomes the major contributor to U.S. supply; energy independence is not achieved until after 2000 because of time delays in capital turnover and constraints on investment, and a world oil shortage in the 1980's brings this problem into national focus
- Continued supply shortfalls, such as the natural gas curtailments in the mid 1970's, will continue well into the future primarily as a result of increasing demand for energy (price regulation keeps prices too low to spur energy conservation) and time delays encountered in bringing on new sources of energy.

**PLANS FOR THE COMING YEAR** — Work will include substantial revision of FOSSIL 1 and formal validation of the model.

### NATIONAL COAL POLICY PROJECT

GEORGETOWN UNIVERSITY  
DOE - \$50,000; Other - \$628,000  
1/77 - 1/78

**OBJECTIVES** — The National Coal Policy Project is bringing together individuals from industry and environmental organizations in an effort to achieve agreement on policy recommendations concerning the increased utilization of coal within economically and environmentally acceptable limits. Although coal is the most abundant domestic fossil fuel resource, the nation has not managed to develop a broad-based understanding of the appropriate role for coal in large part, because of inability to resolve the environmental objections to the burning of coal in a satisfactory manner. Five task forces, composed of equal numbers of environmentalists and industrialists, have been established to study coal-related issues in the areas of: coal mining, coal transportation, air pollution, fuel utilization and conservation, and energy pricing. Discussions are conducted under the "Rule of Reason," whereby agreement is first sought on the relevant "facts" to make possible reasoned, unemotional consideration of the issues. The task forces are directed by the plenary group, the Project's oversight body, composed of the Chairmen and Vice-Chairmen of the Task Forces and the Chairmen of the caucuses of each side.

**RECENT WORK AND ACCOMPLISHMENTS** – The Project met in plenary session 4 times and held more than 30 task force meetings across the country. Many of the task force meetings have included field-trip site inspections, which were useful in acquainting the project participants with local conditions and problems and in providing first-hand knowledge of certain technologies. The approach used has created a rapport among the participants and has been instrumental in facilitating the discussions. The task forces have submitted reports on their respective areas of responsibility to the plenary group, which is currently assembling a project report.

**PLANS FOR THE COMING YEAR** – The first phase of the National Coal Policy Project will be completed with the issuance of a report in April 1978; however, there are several areas where the participants feel that further discussion will produce significant results. A continuation phase of the project is under consideration with plans to include those topics left unresolved at the conclusion of this first phase, as well as subjects not yet considered for which there is a potential for meaningful discussion by the group.

## INTERNATIONAL ECONOMIC ASSESSMENT OF COAL RESEARCH

NATIONAL COAL BOARD  
DOE - \$317,569  
11/20/75 - 3/31/83

**OBJECTIVES** – An economic Assessment Service was set up to carry out studies in the field of coal R&D to assist the member countries in evaluating the application of coal conversion techniques in their respective countries and in formulating new research projects. Basically, the task is to identify promising developments (or obstacles to achievement) where collaboration between member countries could have an important impact on changing the economics of using coal in the future. There are studies also covering the economics of coal supply and trade and the transport of coal or coal-based energy. Liaison is thus maintained with the Reserves and Resources Service, the Mining Technology Clearing House, and the Technical Information Service.

**RECENT WORK AND ACCOMPLISHMENTS** – The program consists of nine studies, four of which are concerned with assessment of coal conversion (and power generation) technology, three with coal supply transport and trade, and one broad study to assess the economics of pollution control in selected areas associated with coal. A final study deals with the relative economics in the marketplace of different coal-based uses, and includes views of the economics of other primary energy sources. In the area of economic and technical criteria for coal utilization plants, four draft reports have been issued to member countries. One on economic conventions, in particular, shows the different rates of return that are required by different countries and organizations, private and public. The other reports compare performance data on gasification and liquefaction processes and on advanced coal-fired power generation systems. Indications are given of the reliability of these data and of the operational problems of individual processes. Development needs are also specified. Substantial progress has been made on reviewing the control of sulphur oxides in combustion. This is the first phase of a wider study on the economics of the environmental aspects of coal utilization. Sulfur contents in world coals are compared in various detail. Limits on SO<sub>x</sub> emissions and ambient standards, as specified in member countries, are reported as are performance and costs of emission control methods, including coal preparation, chemical cleaning, sorbent addition (particularly during fluidized combustion) and flue gas desulphurization. Status of the various technologies and development needs are discussed. Work also continued on a study to assess the likely future cost

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and availability of coal. A report has been issued surveying the large number of plans and projections for coal that have been published by various institutes and governments in the past 2 or 3 years. The Service has also begun work on producing cost models for various significant countries to enable a more specific view to be given on the likely costs and possible prices of coal in the future.

***PLANS FOR THE COMING YEAR*** – The cost models study will be continued into 1978 when, approximately in the middle of the year, an Economic Assessment Service Report giving views on the cost and supplies of coal over the next 25 years is expected to be available. A study will be carried out also on the economics of controlling pollution associated with effluent liquids from gasification plants. In addition, work in these areas will be ongoing: The economics of transporting coal and coal-based energy; economic models of coal conversion; investment and operating costs associated with a major coal conversion plant; and relative costs of coal-based energy, which will integrate these results on the relative costs of converting coal into different energy forms and compare them with the costs of these energy forms and those provided by other energy sources.

### **STRATEGIC BACKDROP ANALYSIS**

**THE FUTURES GROUP**  
**DOE - \$168,000**  
**8/1/77 - Continuing**

***OBJECTIVES*** – The data and analytical framework to produce a strategic backdrop analysis for fossil energy are being developed. Key socioeconomic factors that correlate with end-use energy requirements will be identified to define alternative energy targets through 2025 by end-use sector and fuel mix. Alternative supply and end-use technology combinations that could be implemented to meet these targets will then be defined. Feasible technological scenarios will be evaluated according to economic factors, primary resource implications, and environmental impacts, and conditions highlighted that provide advantages for one competing technology scenario over another. The analytical techniques will be transferred to the Fossil Energy staff in training sessions so that both the substantive data and the methodological structure can be used in internal analysis.

***RECENT WORK AND ACCOMPLISHMENTS*** – Three energy targets have been defined in terms of optimistic, moderate, and pessimistic socioeconomic growth patterns. Disaggregated end-use demands for the residential, commercial, transportation, and industrial sectors have been estimated for each target through 2025, further divided by fuel mix where appropriate. Technology briefs have been written to describe the characteristics of the fossil fuel technologies and conservation techniques to be included in the alternate technology scenarios. Preliminary design work is underway to integrate alternate energy strategy scenarios with the energy targets.

***PLANS FOR THE COMING YEAR*** – Most of the substantive project tasks will be accomplished, including completion of target and scenario integration and preliminary evaluation of alternative energy strategies. Transfer of analytical techniques will also be started.



## ECONOMIC EVALUATION OF FE DEMONSTRATION PLANT PROGRAM

MITRE CORPORATION  
DOE - \$377,000  
10/1/76 - 12/1/77

**OBJECTIVES** – This economic evaluation is assessing the Fossil Energy Demonstration Plant Program and providing new methods to assist in the development of demonstration program strategies. The approach used in the assessment considers the national needs for the technologies that are candidates for demonstration. The development of new methods focuses on the applications of the principles of structured value analysis to the rating of alternative demonstration program strategies. Related supporting studies include projections of capital requirements and availability, timing of the commercialization of alternative fuels, estimates of energy contributions from non-fossil technologies to the U.S. supplies, and near-term availability of technologies for demonstration programs. Results from this study will support decisions concerning the selection of specific demonstration program strategies to meet the nation's energy goals.

**RECENT WORK AND ACCOMPLISHMENTS** – The economic evaluation is in the final stages of analysis and report preparation. Supporting information has been documented in a series of reports covering (1) energy system and energy/economic models, (2) capital requirements and availability to meet future U.S. energy needs, (3) chronology of events relevant to U.S. energy issues, (4) timing of synthetic fuels commercialization, (5) possible contributions of non-fossil energy technologies to the U.S. energy supplies by the year 2000, (6) matching of fossil energy technologies' status with projections of the nation's energy end-user demands, and (7) a methodology for evaluation of alternative fossil energy demonstration program strategies. Evaluation of five demonstration program strategies is in progress and will be included in the final report.

**PLANS FOR THE COMING YEAR** – The work, in the final stages of analysis and report preparation, will be completed.

## IN-SITU COAL GASIFICATION PROGRAM ANALYSIS RESEARCH STUDIES

OAK RIDGE NATIONAL LABORATORY  
DOE - \$150,000  
8/1/76 - Continuing

**OBJECTIVES** – These studies assist the Division of Program Control and Support in its program review function by assessing the potential for commercialization of candidate in-situ coal gasification processes by performing technical and economic evaluations of competing processes and systems.

**RECENT WORK AND ACCOMPLISHMENTS** – Technical and economic evaluations of in-situ coal gasification processes were completed that compared linked vertical well, packed bed, longwall generator, and steeply dipping bed on the basis of a qualitative assessment of their technical merits and their suitability for application to various resource types and configurations in the continental United States. Three seam thicknesses (< 15 ft, 15 to 50 ft, and > 50 ft), two seam dips (< 45 and > 45 degrees), and seam depths to 3000 ft were considered. The United States was divided into four regions, and a predominant or representative coal type was selected within each region for purposes

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of displaying the processes to their best advantage. Russian experience, where it applies to the regional conditions under consideration, is also described. Conceptual process designs and cost estimates were prepared for three facilities, illustrating the following potential applications of underground coal gasification: a 900-Mw<sub>e</sub> combined-cycle electric generating plant fueled by low-Btu gas, a substitute natural gas (SNG) plant producing 155 MMscf/d of a 954-Btu/scf gas, and a synthesis gas (Syngas) plant producing 388 MMscf/d of gas suitable for further chemical conversion. In-place coal consumption for the three facilities is 18,073, 22,951, and 22,951 t/d, respectively, based on a subbituminous Wyoming coal. Respective capital investments were estimated to be \$395, \$351, and \$312 million in first-quarter 1977 dollars. Calculated overall thermal efficiencies for the three facilities were 24, 38, and 40 percent, respectively, based on in-place coal. Designs used experimental data obtained at the Laramie Energy Research Center on the linked-vertical well in-situ coal gasification process. Product prices were calculated as a function of the debt/equity ratio, the annual earning rates on debt and equity, the cost of coal, and plant factor (on-stream efficiency). On the basis of a debt/equity ratio of 70/30, an interest rate on debt of 9 percent, an aftertax earning rate on equity of 15 percent, a coal feed cost of \$10/ton, and a plant factor of 90 percent, product prices were 24 mills/kwhr for electricity, \$3.66/10<sup>6</sup> Btu for SNG, and \$3.20/10<sup>6</sup> Btu for Syngas.

**PLANS FOR THE COMING YEAR** – A conceptual design and cost estimate will be performed for a commercial in-situ coal gasification plant (including above-ground facilities) for the production of approximately 15,000 bbl/d of gasoline via methanol.

## MANAGEMENT PLAN FOR ENHANCED OIL RECOVERY

LEWIN AND ASSOCIATES, INC.  
DOE - \$136,000  
3/76 - 1/77

**OBJECTIVES** – The purpose of this effort was to provide technical assistance, data gathering, and economic modeling support to DOE's Division of Program Control and Support in preparing their *Management Plan for Enhanced Oil Recovery*. The management plan set forth DOE's 5-year R&D plan in enhanced oil recovery (EOR) including the relative priorities, funding levels, and timing for research projects.

**RECENT WORK AND ACCOMPLISHMENTS** – Six major products were to be delivered as part of this effort: develop measures of effectiveness for Federal RD&D programs in EOR; recommend high-priority R&D targets in EOR and define their budgetary and timing implications for DOE; estimate the cost-effectiveness of possible Federal R&D strategies; provide analytic support for the preparation of the EOR Management Plan; define the supplementary analyses required for effectively implementing the EOR Management Plan; and incorporate critiques and comments on the data base and models used for the study. The study culminated with a three-volume report prepared for DOE entitled, *Research and Development in Enhanced Oil Recovery*.

**PLANS FOR THE COMING YEAR** – Additional resource data and field test results are being incorporated to update the report.

## **SUPPORT SERVICES TO FOSSIL ENERGY**

**COMPUTER SCIENCES CORPORATION**

**DOE - \$4,500,000**

**3/77 - 2/80**

**OBJECTIVES** – Project management support, systems development, management analysis, and computer system development support are being provided to the Divisions of Program Control and Support, Coal Conversion, Magnetohydrodynamics, and Power Systems.

**RECENT WORK AND ACCOMPLISHMENTS** – Specific management support projects were performed in the following areas: project management system planning, development, integration, and implementation; computer systems requirements determination, design, development, and installation; management analysis; and management consulting. Major accomplishments were the development and installation of the contract file card, uniform contractor reporting guidelines, and systems review and analysis. Computer systems developed, implemented, and enhanced at the staff and division levels were the Procurement Planning Status Reporting Systems, Fossil Energy Data Management System, and Financial Technical Planning Information System. Supporting management operation manuals and system operation and procedures, incorporating budget formulation and execution and procurement administrative requirements, were also developed. Management information was concentrated in a single document that collated and consolidated budget and procurement planning and execution data with contractor performance data, and an overall analysis of each contract. Special studies and analyses were performed upon request primarily in the budget execution, procurement administrative processing, and financial areas. Assistance was also provided in formulating the budget and development of background data.

**PLANS FOR THE COMING YEAR** – Work will continue in the development of program and project management systems, management consulting, and computer systems analysis and programming. Emphasis will be placed on the enhancement of existing systems and their extension into the Fossil Energy organization. System integrity, compatibility, and interaction will be the major objective of all system development and implementation.

## **SUPPORT SERVICES TO FOSSIL ENERGY**

**ENERGY AND ENVIRONMENTAL ANALYSIS, INC.**

**DOE - \$1,997,909**

**4/76 - 6/78**

**OBJECTIVES** – A wide range of support services is being provided to Fossil Energy. Areas of support include planning/prioritization of RD&D, technical evaluation of FE technologies, and environmental analysis of FE projects.

**RECENT WORK AND ACCOMPLISHMENTS** – Major accomplishments in the area of RD&D planning/prioritization include the development and demonstration of a methodology for estimating the benefits of various FE research programs. This methodology has been applied to high-Btu gasification and coal liquefaction technologies. EEA also has developed an industrial sector analysis that incorporates risk in financial feasibility analysis concerning investments in large-scale technologies. It also incorporates the effect of FE RD&D funding on the financial outcome profile. It simulates how large corporations using state-of-the-art investment analysis might view an

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investment in coal liquefaction. The analysis also captures in the revenue stream from large-scale plants the revenues from highly valued chemical products. The industrial sector data bases were surveyed; sample screening approaches and methodologies for industrial sector analysis were developed. Estimates of coal prices through 2020 were provided for use in agencywide research program review, a systematic approach was developed to identify and characterize all the elements in coal use from mining to ultimate energy utilization, and significant elements in the coal cycle not addressed by current programs or study were identified. The coal cycle was analyzed from an economic point of view to determine whether there are opportunities for maximizing the efficiency of various combinations of coal preparation, transportation, destination, and handling. EEA also has developed an assessment method that was used to compare alternative coal scrubbing and beneficiation methods in terms of cost to the user and the extent to which national coal resources can be used.

Environmental analysis work included gathering alternative pollution control data to be used in reviewing draft EPA coal gasification air emission standards. Additionally, EEA provided the technical and planning support necessary to complete the Coal Gasification Environmental Development Plan, established protocols and procedures for subsequent plan preparation, and developed an environmental and safety program for coal gasification that reflects a concern for environmental priorities, the timing of technology development, and the balance of work responsibilities within DOE. Finally, an environmental evaluation of the FY 1979 budget was conducted. Issues requiring resolution through work in FY 1979 were identified, suggested environmental compliance activity proposals were developed, and issue papers on a wide range of environmental areas influencing the FY 1979 budget were prepared.

***PLANS FOR THE COMING YEAR*** – Work for the coming year will focus on further development and expansion of the industrial sector technology use model including development in three primary areas: more detailed industrial energy demand specification; more careful technology cost specification; and more extensive development of program logic. In the environmental area, work will include review and analysis of environmental data on pipeline and fuel gas demonstration plants and support of the preparation of EIA's and EIS's as necessary; preparation of environmental research plans for direct combustion, magnetohydrodynamics, and advanced power systems; data collection on air and water emissions characteristics of FE technologies; and assessment of EPA regulatory actions in these areas.

## **FOSSIL ENERGY PROGRAM SUPPORT**

**SDC ENERGY AND RESOURCES DEPARTMENT**

**DOE - \$3,644,590**

**4/76 - 6/79**

***OBJECTIVES*** – A broad spectrum of technical and administrative support services were provided in 1977 that resulted in guidance and review for the development of new technology using fossil energy with coal as the principal resource.

***RECENT WORK AND ACCOMPLISHMENTS*** – The support provided to DOE has resulted in a series of reports providing technical reviews of elements of the Fossil Energy Program, several data acquisition/management information systems, technoeconomic studies of selected processes in the

research phase, and various consulting services. Major areas of support included the following. Work on the Performance Assurance System (PAS) includes reliability, availability, quality assurance, configuration management, and life-cycle cost analysis. SDS is planning and assisting in the Synthane Pilot Plant reliability demonstration scheduled late in 1978. SDC has provided technical support in the PAS area to the national laboratories and FE project managers. SDC, in conjunction with the NBS, has developed and implemented the Fossil Energy Equipment Data System (FEEDS) to centralize, collect, and analyze fossil energy failure and operability data.

Administrative support to the coal program included analysis of procedures for proposal and financial tracking, and assistance in the implementation of an improved tracking system. In support of the coal utilization program, the national research capability and needs for innovative research in the combustion area were studied; for synthetic fuel programs, evaluations were provided of selected processes in the advanced bench scale and research phase. Specific topical studies were also prepared in the areas of fluidized-bed combustion, the effects of scaling in combustion experiments, the prospects for nuclear instrumentation in coal analysis, the problem of hot gas cleanup in synthetic fuel plants, and the status of flue gas desulphurization. Engineering and construction support services were provided for the "Gasifiers in Industry" demonstration program for low-Btu coal gasification. Services included third-party design review, and monitoring of engineering and construction schedules and costs.

A project monitoring and reporting system was developed, with SDC assistance, to be utilized by the Assistant Director for Oil and Gas to make administrative and technical management decisions, conduct program and project status reviews, provide state-of-the-art program and project monitoring and control through technical and financial briefings, and identify technology transfer capabilities on a project-by-project basis within the program. A second effort for OGSIST resulted in identification of users and user needs for information relevant to offshore oil and gas resource development (exploration, development, production, transportation, and onshore impacts). Data sources, acquisition methods, and contents were identified. The completeness of data to satisfy requirements and to develop a plan for DOE involvement to assist the users in a useful format was assessed.

**PLANS FOR THE COMING YEAR** – The team of engineers and scientists forming the Energy and Resource Department will continue to support the FE research efforts in the development of new technology, including work on PAS and the Synthane Pilot Plant reliability demonstration.

#### **TECHNICAL SUPPORT FOR FOSSIL ENERGY PROGRAM**

**BOOZ, ALLEN & HAMILTON, INC.**  
**DOE - \$2,240,469**  
**4/13/76 - 7/13/79**

**OBJECTIVES** – This contract provides for planning, analytical, and technical support to the Fossil Energy (FE) staff and to the offices of the several program areas. This effort includes data gathering and analysis, program development, program issue analysis, program and project reviews, plan documentation and reporting, general staff assistance, and special assigned studies. In support of the technical development program, these services contribute to FE headquarters decision-making process through qualitative and quantitative analyses, particularly in programmatic and budgetary areas.

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**RECENT WORK AND ACCOMPLISHMENTS** – Assistance was provided to the Division of Program Control and Support (DPCS) in analyzing, designing, preparing, and publishing the *Fossil Energy R&D Program (FY 1978)* report. A companion to that budgetary and planning document is the *Fossil Energy Program Report (July 1975 - October 1976)*, which documents over 450 FE contracts. Booz, Allen reviewed the contractor submissions, and designed and prepared the final copy of this widely distributed document in coordination with FE Senior Staff. Seven Management Review and Control Documents (formerly Program Approval Documents) were also developed and processed through several levels of review from materials provided by DPCS staff and the several technical divisions.

For the Division of Oil, Gas Shale and In-Situ Technology (OGSIST), a detailed technical and environmental evaluation was performed of all major technologies being developed for processing oil shale, and results published in a five-volume report. Another task, part of the 5-year Management Plan for Underground Coal Gasification, involved a study providing detailed documentation of the engineering and environmental aspects of subsidence expected to result from the in-situ combustion of western coal beds. Booz, Allen continued its role as the coordinating contractor in developing the Enhanced Oil Recovery Technology Implementation Plan (TIP). This task involves technical, logistical, and coordinating support plus attendance at working groups on various processes and synthesis of the information developed. A recommended strategic plan for the Enhanced Gas Recovery Program is being developed describing and quantifying major policy issues on the basis of background information collected from industry. In concert with these industry interviews, an analysis of undiscovered conventional natural gas reserves and subsequent production was conducted at a regional level using a discounted cash flow model and historical data. The computer model was implemented on DOE systems for future availability.

In support of the Division of Major Facility Project Management (MFPM) Booz, Allen developed standard criteria using discounted cash-flow principles for the economic evaluation of alternative pipeline and fuel gas plant projects. Economic analyses of four alternative approaches to coal conversion commercialization have been made also; since results were close, consideration of risk and other nonfinancial issues is being considered. The ability of the AFBC R&D program to produce a demonstration plant by FY 1982 has been assessed, including identification of major activities, technical problems and resulting research projects, and relative timing. A report collation service for MFPM program management, involving collection of data into overall project reports at varying levels of detail, has been performed.

**PLANS FOR THE COMING YEAR** – Several ongoing tasks will be completed, such as the provision of support in the area of strategic planning and analysis of mathematical models. Also, Program Plans covering a 5-year period for several of the major technology areas will be designed and prepared for publication. Work on the seven MRCs will be concluded, and a new, larger version (FY 1977) of the FE Program Report will be prepared for issuance. The natural gas reserves analysis and its relevance to the Enhanced Gas Recovery Management Plan will receive an intensive effort. The three-volume 5-Year Program Plan for In-Situ Coal Gasification will be updated and refined, and data gathering and process evaluation performed.

## **SUPPORT SERVICES TO FOSSIL ENERGY**

**TRW ENERGY SYSTEMS GROUP**  
DOE - \$6,467,000  
3/76 - 7/79

**OBJECTIVES** – Support services are being provided to various organizations within DOE Fossil Energy. This work includes data collection and analysis, program development, analysis of program issues, project management support, program and project review, plan documentation and presentation, special studies, and general staff assistance.

**RECENT WORK AND ACCOMPLISHMENTS** – Technical evaluations, data correlations, and design option development to assist program planning and analysis were provided in the areas of coal conversion, resource recovery and environment, health and safety. Significant accomplishments in coal conversion include a fluid-bed char gasifier process design, coal conversion plant cost escalation study, and evaluation of heat pipes for coal gasification. Planning and analysis activities included supporting the ERDA National Plan (ERDA 77-1); development of Program Approval Documents (PADS) for Petroleum and Natural Gas, Magnetohydrodynamics, Coal Conversion, and Coal Utilization; a Program Plan for Fossil Energy Demonstration Plants; Coal Gasification and Liquefaction R&D Plans; the Coal Gasification Program Management Plan; and the Coal Conversion Environmental Development Plan. Data from the Wilsonville and Tacoma SRC plants were analyzed, and they showed that both plants give comparable data when using the same coal type. TRW visited the various liquefaction pilot plants, prepared a report of the material balance procedures used by the various liquefaction pilot plant operators, and arranged a meeting of the various contractors to exchange technical information and comments on their material balance techniques. In the area of resource recovery, significant accomplishments included a report outlining the requirements for commercialization of in situ coal gasification technology, a Preliminary Project Plan for the Western Gas Project, economic analysis of natural gas stimulation applied to the eastern devonian shales, and the Western Gas Sands Project Implementation Plan.

**PLANS FOR THE COMING YEAR** – Previous efforts in support of the Division of Magnetohydrodynamics will be continued with emphasis on program planning. Additional tasks for other Fossil Energy organizations will be defined as required by DOE.

## **SUPPORT SERVICES TO FOSSIL ENERGY**

**SCIENCE APPLICATIONS, INC.**  
DOE - \$620,302  
5/25/77 - 5/24/79

**OBJECTIVES** – Broad analytical, technical, planning, and management (special projects) support is being provided to the Division of Program Control and Support (DPCS) in the areas of environment, socioeconomics, and occupational safety and health. This support includes independent review of Fossil Energy RD&D projects to assure that projects are responsive to national objectives and regulations concerning the environment, safety, and health. Secondary support to other FE offices and divisions may be authorized by DPCS to assure proper completion and adequacy of overall environment and safety efforts.

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**RECENT WORK AND ACCOMPLISHMENTS** — Two major documents were prepared recently, the Environmental Development Plans (EDP's) for Oil Supply and Enhanced Gas Recovery. A number of documents prepared by others on occupational safety and health of the fossil fuel demonstration sites of DOE were critically reviewed and revised prior to becoming official Government publications. Support in short-turnaround environmental studies, analyses, and assessments for DPCS is being provided regularly.

**PLANS FOR THE COMING YEAR** — Field visits to fossil energy projects will continue to assist DPCS in assessing the environmental, safety, and health performance of these operations. Preparation of documents, reports, and briefings on environmental, safety, and health aspects of the DOE program for mining and preparation of solid fuels (coal and oil shale) is expected to be a key support area.

### PROJECT REVIEW PROGRAM SUPPORT

TETRA TECH, INC.  
DOE - \$175,268  
5/25/77 - 5/24/78

**OBJECTIVES** — Technical support services are being provided to the Assistant Secretary of Energy Technology with formal verification of project performance. Because of the wide range of technology associated with the various Fossil Energy projects, the services of eminent consultants are used to assist in performing the individual project reviews of the various technical processes and related facilities. Each project review involves a literature search for relevant technical information; assembly of a data package that provides background information for the reviewers; use of appropriate consultants; preparation of a draft report after the on-site review, incorporating team comments as well as consultant findings; and preparation and publication of the review under the direction of the DOE review chairman.

**RECENT WORK AND ACCOMPLISHMENTS** — Progress reviews of seven Fossil Energy projects have been performed. Three of these projects are coal-related: Pope, Evans and Robbins' development of a multicell fluidized-bed boiler; Morgantown Energy Research Center's development of longwall generator techniques for gasifying coal underground; and Pennsylvania State University's research on predicting the behavior of a coal in preconversion processing. The other projects were: Bartlesville Energy Research Center's Project Halo research program to develop and synthesize more effective and economical ways of exploring for petroleum and natural gas; Phillips Petroleum Company's development of a micellar flooding process for tertiary oil recovery; Penn Grade Crude Oil Association's evaluation of the Maraflood micellar-polymer process for tertiary oil recovery, and the Dow Chemical Company's investigation of techniques for in-situ recovery of gas and oil from Michigan Antrim oil shale.

**PLANS FOR THE COMING YEAR** — Assistance will be provided to DOE on a project review of Petroleum Technology's chemical explosives fracturing process for enhanced gas recovery in shale and sand formations and with other project reviews as assigned.



## SYSTEMS STUDIES SUPPORT

BROOKHAVEN NATIONAL LABORATORY  
DOE - \$170,000  
7/1/75 - Continuing

**OBJECTIVES** – Analytical support is being provided to the Long-Range Strategy function of DOE's Division of Program Control and Support. This support involves the continuing assembly, qualification, updating, and application of data bases and analytical tools that describe the technical, environmental, and economic characteristics of fossil energy technologies and resources. When required, the program includes the relationship of fossil energy to other energy sources, to the U.S. economy and international trade, and to institutional constraints.

**RECENT WORK AND ACCOMPLISHMENTS** – Data required by the Regional Energy System for the Planning and Optimization of National Scenarios (RESPONS) model, developed by the Bechtel Corporation, were obtained and documented. Several runs were made using the improved parameters, and analyses of the results performed. The matrix generator was reprogrammed in a language compatible with and available on BNL's CDC-7600. Modeling improvements and refinements were recommended.

**PLANS FOR THE COMING YEAR** – Surveying of available analytical models and data bases will continue. When appropriate, they will be evaluated in detail and made accessible for use in strategic planning. Applications of the RESPONS model and others will be made as needs dictate.

## REGIONAL APPLICATION OF FOSSIL ENERGY TECHNOLOGIES

SYSTEMS CONSULTANTS, INC.  
DOE - \$94,809  
6/28/76 - 5/25/77

**OBJECTIVES** – A methodology is being developed that relates fossil energy technologies to specific regional energy problems. Indices are prepared to identify and scale critical regional energy characteristics, and fossil energy program technologies are quantified to permit a comparison between technology requirements and regional resources. A specific analysis is performed that demonstrates the feasibility and application of the methodology to energy policy analysis.

**RECENT WORK AND ACCOMPLISHMENTS** – Energy Descriptive Indices have been developed that aid in defining the energy characteristics of a region. The importance of the index values is that they are sensitive to the mix and amount of energy fuels consumed. A format has also been developed facilitating the comparison of fossil energy technologies with regional resources and characteristics. Two methodological approaches have been devised that provide a sound analytical basis for relating fossil energy technologies to specific regional energy problems. A specific analysis is performed that applies low-Btu gasification to the east north central region to help in reducing the consumption of natural gas by industry.

**PLANS FOR THE COMING YEAR** – This regional energy study will be updated and the methodology refined in a separate contract sponsored through Brookhaven National Laboratory. In addition, several energy policy analyses will be accomplished not only to test the methodology but to demonstrate its flexibility and ease of application. The analyses selected will be consistent with the goals of the National Energy Plan.

## MAPPING PROJECT ON ENERGY AND THE SOCIAL SCIENCES

YALE UNIVERSITY INSTITUTION FOR SOCIAL AND POLICY STUDIES

DOE - \$155,000  
10/1/76 - 9/30/77

**OBJECTIVES** – This project will study the possible contributions of academic social science research to specific social problem areas resulting from energy supply shortages and transitions to new energy supply and conversion technologies. The project seeks the following specific objectives: (1) to identify, classify, and analyze relevant social science research publications, studies in progress, and other concepts and methodologies, (2) to create a national clearinghouse for energy and social science publications and reports, (3) to prioritize possible social science research topics in energy, (4) to consider the relevance of social science research in energy to the development of policy in the field, (5) to create opportunities for interactions between academic social scientists and DOE representatives, and (6) to involve members of the broader social science research community in energy research.

**RECENT WORK AND ACCOMPLISHMENTS** – Progress has been made toward identifying the useful existing research and specifying needed new research in several areas, including energy and social organization; energy boomtowns, the diffusion of innovations, public participation, regulatory systems, and energy survey data. A national clearinghouse for research information on selected energy topics is being established. Workshops are being conducted and other interactions established with DOE. The process of mapping, the systematic identification of a research agenda, is being studied with the objective of developing guidelines for future efforts.

**PLANS FOR THE COMING YEAR** – The major thrust of the Mapping Project work this year will be to develop a methodology to compare the social impacts of different fuel cycles. Prioritization of various research topics will continue, as will the development of the National Clearinghouse for Energy and the Social Sciences. The Clearinghouse will establish a set of standardized subject categories useful for social science investigation and compatible with DOE's information systems. Acquisition work will continue and indexing will begin. A Quarterly Review of Energy and the Social Sciences, to be issued this year, will summarize the results of the project for DOE personnel and social scientists outside DOE who are following this work. Emphasis will be on suggestions of priority for DOE social science and policy research.

## ENERGY MODELS AND DEPLETABLE RESOURCE PRICE-QUANTITY RELATIONSHIPS

DECISION FOCUS, INCORPORATED

DOE - \$194,999  
4/19/77 - 11/30/77

**OBJECTIVES** – The research objectives were to determine the adaptability and utility of the SRI-Gulf National Energy Model in assessing the potential market impacts of the full range of ERDA technologies; identify and implement the near-term changes in data, logic, constraints, and assumptions that would more nearly reflect the consensus of the ERDA Program Planning Study Working Groups and Analysis Committee; and conduct research and analyses of the potential market impacts of the results of alternative programs of RD&D and nontechnical Federal initiatives and policies.

**RECENT WORK AND ACCOMPLISHMENTS** – The evaluation was undertaken by making selected runs on the SRI-Gulf model, carrying out a careful review of its structure and detailed assumptions, and construction of small-scale generalized equilibrium models to test the sensitivity to specific modeling assumptions. They included the well life and decline profile of depletable resources such as oil and gas and the impact of price regulation. The conclusion of the evaluation was that the existing SRI-Gulf Energy Model was of limited usefulness in supporting a budgetary exercise such as MOPPS. It lacks detail in some areas, has too much detail in others, and lacks the flexibility to adapt specific portions of the model to match the assumptions used by the supply and demand working groups of MOPPS. While the generalized equilibrium modeling approach holds potential as an integrating framework, a more flexible, user-oriented computer implementation is needed to realize this potential.

**PLANS FOR THE COMING YEAR** – The project has been completed, and the final report was submitted for review to DOE in November 1977. A final version of this report will be issued in February 1978.

## **COAL AND GAS PIPELINE FEASIBILITY STUDY**

**SYSTEMS CONSULTANTS, INC.**

DOE - \$162,891

9/5/77 - Continuing

**OBJECTIVES** – This study is determining the technical and economic feasibility of using a coal and gas pipeline system to bring West Virginia coals of medium- and high-sulfur content to U.S. East Coast utility markets. Coals, whose sulfur and mineral matter content rendered them unacceptable for current or projected steam coal markets, are to be beneficiated to yield a low-sulfur low-ash fraction. The middlings product is to be gasified to produce synthesis gas. The synthesis gas is cleaned and subsequently used to transport the clean coal, via a pipeline, to East Coast markets, such as the Philadelphia area. The overall system will provide the end-point user, such as a utility, with clean fuel and the flexibility required to meet a varying demand without the need for expensive stack-gas cleaning equipment.

**RECENT WORK AND ACCOMPLISHMENTS** – The initial effort consisted of selecting and evaluating the design criteria. A mine mouth plant is to be designed to take a typical West Virginia coal and, by unit processes of beneficiation, gasification, and pneumatic (synthesis gas) transport, supply the fuel needs of a 1000-Mw electric power plant located in the Philadelphia area. The product flue gas from the power plant is to meet the local source emission standards without the need for flue gas desulfurization equipment.

**PLANS FOR THE COMING YEAR** – The study will continue with a system design and optimization phase. The beneficiation subsystem is to remove most of the pyritic sulfur from the coal. One of two gasifiers—an agglomerating fluidized-bed or a two-stage entrained-flow slagging type—is to be selected and will operate at 500 psi or less. The gas and clean coal are to be transported by a 36-in. pipeline a distance of 300 miles.

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## ENVIRONMENTAL GUIDELINES FOR FOSSIL ENERGY SITES

PITTSBURGH ENERGY RESEARCH CENTER  
DOE - \$275,000  
1977 - Continuing

**OBJECTIVES** – Environmental guidelines are being developed and criteria reviewed for site evaluation, analysis, and selection for coal conversion processes. The guidelines, developed as a function of the technology, would highlight the significant environmental impacts and resource needs of one process versus another, based on specific site conditions.

**RECENT WORK AND ACCOMPLISHMENTS** – Detailed project planning efforts were completed, which resulted in the preparation of an expanded work plan for future contractor efforts. Reviews of RFP submissions indicated that the proposals were funded into two categories: those with high levels of understanding of the coal conversion and utilization technologies and low levels of knowledge in the socioeconomic areas and vice versa. Selection and finalization of the contracts occurred in September. Subsequent monthly meetings with the contractors have produced draft chapters of the guidelines document.

**PLANS FOR THE COMING YEAR** – The final products of this phase of the work are scheduled for delivery by July 1978. These products include the text that will provide the data base and criteria and user's guide on the methodology developed for the site evaluation process. Followup work will deal with case studies and methodology refinement. A number of briefings and symposia will be conducted.

## ENGINEERING AND ENVIRONMENTAL ASSESSMENTS

ARGONNE NATIONAL LABORATORY  
DOE - \$47,000  
1976 - Continuing

**OBJECTIVES** – Engineering support will be provided, on an as requested basis, to DOE. This support will consist of quick response reviews of plant designs as well as engineering assessments of operational, environmental, and safety considerations relevant to ongoing tasks. Present tasks in DOE include demonstration plants for low-Btu gasification, liquefaction, and fluidized-bed combustion to be constructed in the near future; successful operation of the demonstration plants; and engineering evaluation of commercial coal conversion plants. This program will provide the Office with needed engineering support in meeting these responsibilities.

**RECENT WORK AND ACCOMPLISHMENTS** – Engineering services were provided to the technical assistant for initial operations by ANL through a consulting subcontract with H.M. Mittelhauser Corp. Some of the specific technical contributions made to DOE projects are: (1) assisted in preparing the official DOE position on EPA proposed standards for high-Btu gasification, (2) reviewed the Coalcon Environmental Assessment Report and recommended additional work, (3) assisted Oak Ridge National Laboratory in preparing environmental monitoring guidelines for demonstration plants, (4) provided guidance to the Conoco, ICGG, and Powerton project managers on EPA regulations affecting these plants, and (5) recommended ambient air testing programs for demonstration plants. A DOE task force evaluation of the impact on emerging coal conversion

technologies of the proposed revision of the New Source Performance Standards (NSPS) was described in a report on Solvent Refined Coal (SRC-1). A report by Mitre and Gilbert which evaluated the NSPS impact on the fluidized-bed combustion of coal was reviewed. Preliminary drafts of the position paper on the proposed NSPS written by the Environmental and Socioeconomic Program Division were reviewed.

**PLANS FOR THE COMING YEAR** – The primary effort will be to conduct engineering reviews of process designs and environmental control technology performance as requested by DOE. In support of that objective and in order to ensure the timeliness of the reviews, a reference library will be established for each potential demonstration program and will be evaluated to ensure its completeness for process design purposes.

## **ENVIRONMENTAL IMPACTS: WATER-RELATED SITE AND PLANT DESIGN CRITERIA**

### **WATER PURIFICATION ASSOCIATES**

DOE - \$92,500

7/1/76 - 3/31/78

**OBJECTIVES** – The feasibility of siting specific coal conversion plants in the coal bearing regions of the United States as a function of the local environmental impacts that can be expected from water-related site, process, and plant design criteria is being determined. The work involves detailed studies of 40 to 50 plant-site combinations in the central and eastern coal bearing regions, and 40 to 50 plant-site combinations in the western coal-bearing regions. The general site and process criteria expected to be derived are: range of water requirements and the conditions for narrowing the range and optimizing the use of water; ranges of residual solid wastes and drainage runoffs, their quantity and nature, and the conditions for narrowing the ranges and minimizing the disposal and drainage problems; localities where local water-related environmental impacts are large, moderate, or small; localities where some processes are more suitable than others to minimize local water-related environmental impacts; and rank-ordering in importance of the site and process criteria themselves in estimating local environmental impacts at individual sites.

**RECENT WORK AND ACCOMPLISHMENTS** – Detailed plant designs were completed for the HYGAS, BIGAS, Lurgi, Synthane, Synthoil, and Solvent Refined Coal processes using various combinations of lignite and bituminous coals. Thirty-three sites were selected in the states of Alabama, Illinois, Indiana, Kentucky, Ohio, Pennsylvania, and West Virginia based primarily on the availability and type of coal, type of mining (underground or surface) and availability of sufficient surface water and groundwater. Water requirements and residual quantities were determined for all plant-site combinations in the central and eastern regions. Detailed energy balances have been made for each of the plant-site combinations to determine the cooling water requirements. The economics of using combined wet/dry cooling and the quantity of water used for cooling have been determined for every plant-site combination.

Under subcontract, Resource Analysis, Inc., defined the reliable surface water and groundwater sources for each site together with the water quality data and provided a general assessment of potential environmental impacts of the energy development facilities. Sites in the central and eastern regions were identified as having sufficient, reliable surface supplies. In these cases, a single water plant water demand was taken to be less than 1 percent of the 7-day, 20-year low flow. Other sites were identified where the surface water supplies are much less reliable, and a

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single coal plant demand would represent a very significant portion of the seasonal low flow. Similar studies were carried out for 50 plant-site combinations in the western region under an Environmental Protection Agency contract. These results are being incorporated into the DOE study to estimate local environmental impacts in all of the major coal-bearing regions of the United States.

**PLANS FOR THE COMING YEAR** – This study will be terminated in 1978. A draft of the final report has been prepared, and a final report is being readied for publication.

## **COAL CONVERSION ENVIRONMENT AND CONTROL ASSESSMENT**

**PITTSBURGH ENERGY RESEARCH CENTER**

**DOE - \$150,000**

**1977 - Continuing**

**OBJECTIVES** – This project is evaluating developing coal conversion processes with respect to their environmental impacts and assessing the applicability of control methods for achievement of environmental goals. Areas will be identified where additional data are required to assess the applicability of control technology and preliminary cost comparisons will be established between appropriate alternative control methods.

**RECENT WORK AND ACCOMPLISHMENTS** – Literature studies have been conducted to determine the available base of information on the characteristics of discharge streams, especially water streams, in coal conversion processes. Both first-generation and advanced coal-gasification/liquefaction processes have been considered. Performance characteristics of control equipment applied to coal conversion operations have been evaluated. Comparisons of the characteristics of coal conversion process streams with contaminated streams from other energy industries have been performed. Data have been gathered on the control levels achievable in these energy industries through the application of various alternative methods. To enhance the environmental studies being performed, a plan for the development of data from an existing commercial coal-gasification facility has been developed. Visitation to the site was completed, and arrangements are being made to follow up with experimental studies. The capabilities of university organizations to provide support to this project have been reviewed and evaluated. This fixed-bed, low-Btu gasification facility should pose a most severe treatment problem.

**PLANS FOR THE COMING YEAR** – For water-quality control, characterization and treatment studies will be performed on an existing low-Btu coal-gasification facility. These data will be used in the assessment of a potential worst-case system for the application of control technology. A report will be written on the evaluations of the water treatment aspects of coal gasification and liquefaction processes. Studies will be initiated on detailed comparison of the economics of alternative control systems for meeting the process and environmental control requirements. These studies will determine the potential for cost savings in the water-treatment portion of coal conversion operations.

## ENVIRONMENT AND ENERGY CONSERVATION SUPPORT SERVICES

PITTSBURGH ENERGY RESEARCH CENTER  
DOE - \$75,000  
1977 - Continuing

**OBJECTIVES** – Support in the areas of environment and energy conservation is being provided, with emphasis on document preparation and review of these programs.

**RECENT WORK AND ACCOMPLISHMENTS** – Support services have included providing essential support services for data review, providing assistance in planning and implementing the FE program, and coordinating activities between Headquarters and the field offices; participating on the Source Selection Board for the selection of a proposal for the development and application of a methodology for monitoring social and economic impacts of demonstration projects; participating on the Fossil Energy Environmental Advisory Board for PON-8, Coal Liquids Refinery Facility; and reviewing and evaluating various FE program documents generated within DOE as well as other Federal agencies; Program Approval Documents (PADs), Environmental Development Plans (EDPs), environmental assessments (coal programmatic), annual reports, speciality reports, proposed programs, and general proposals.

**PLANS FOR THE COMING YEAR** – Work in various areas of environmental planning and development of new environmental research programs will be supported including preparation, and support in the preparation, of Environmental Impact Assessments and Environmental Impact Statements; assistance in the annual updating of EDPs; review for comment and possible modification of environmental statements concerning research in the fields of environmental control and energy conservation; and coordination of activities performed by FE and its field organizations (with their respective contractors) pertaining to environmentally oriented projects and programs.

## ENERGY CONSERVATION IN COAL CONVERSION PROCESSES

PITTSBURGH ENERGY RESEARCH CENTER  
DOE - \$150,000  
1976 - Continuing

**OBJECTIVES** – This project is developing an energy requirement survey methodology for coal conversion processes to identify and document energy conservation opportunities and to provide guidelines for selection, analysis, and implementation of energy conservation techniques.

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presented at the International Conference. A Special Research Support Agreement (SRSA) between PERC and Carnegie-Mellon University was carried out to explore the potential for energy conservation in the C.F. Braun conceptual commercial Synthane process.

**PLANS FOR THE COMING YEAR** – A first- and second-law efficiency analysis of the major unit operations and the overall system will be performed on the C.F. Braun conceptual commercial Synthane process. This work will be accomplished through a SRSA with Marquette University. The SRSA with Carnegie-Mellon University will be renewed with the objective of continuing the characterization of the potential for energy conservation in coal conversion to liquid and gaseous forms and in direct utilization of coal.

## **COAL CONVERSION PROCESS WASTES: ENVIRONMENTALLY ACCEPTABLE DISPOSAL**

**PITTSBURGH ENERGY RESEARCH CENTER**  
DOE - \$80,000  
1977 - Continuing

**OBJECTIVES** – Environmentally sound waste-disposal procedures are being developed for bench, PDU, and pilot-scale coal conversion process wastes. Based on these procedures, potential waste-handling problems and solutions for large-scale or commercial facilities will be identified.

**RECENT WORK AND ACCOMPLISHMENTS** – Work has been initiated to obtain qualitative and quantitative data to characterize liquid and solid wastes generated at the PERC site from ongoing coal conversion process research. The characterizations include physical, chemical, and toxicological properties and/or expected disposal alternatives. Bench-scale apparatus has been assembled to develop a preliminary experimental protocol to determine the leaching potential of solid residuals.

**PLANS FOR THE COMING YEAR** – Visits to other energy research facilities and/or coal conversion pilot plant facilities are planned to determine the feasibility of transferring or modifying any handling or disposal techniques developed in this research. Studies are planned to assess residual toxicities from representative materials, and the following new initiatives will be undertaken: examine potential for volume reduction of wastes when generated and when collected, alternative waste-collection techniques, alternative disposal techniques for waste at the various stages of treatment, and recyclability of the wastes generated; and prepare report, based on the PERC waste study, that will provide waste-handling information and recommendations with economic and environmental considerations highlighted.

## **ENVIRONMENTAL CONTROLS FOR LOW-BTU GASIFICATION**

**OAK RIDGE NATIONAL LABORATORY**  
DOE - \$180,000  
8/15/77 - Continuing

**OBJECTIVES** – This project is collecting and evaluating the technical and the economic information regarding environmental control technologies that may be applicable to low-Btu coal gasification processes.



**RECENT WORK AND ACCOMPLISHMENTS** — A detailed work plan for the project has been developed that identifies the gasification processes and environmental control technologies of interest in this study.

**PLANS FOR THE COMING YEAR** — The technologies identified will be reduced to a representative list of 25 environmental control technologies and up to 4 representative gasification processes that will be subjected to more detailed study in the second phase of this project. This detailed study will consist of developing the characteristics, costs, and energy requirements of each of the 25 representative environmental control technologies. In addition, costs and energy requirements are proposed to be developed for eight selected case studies wherein the representative low-Btu gasification processes will be integrated in conceptual plants with several of the representative environmental control technologies.

## ENVIRONMENTAL ASSESSMENT OF THE PERC SITE

PITTSBURGH ENERGY RESEARCH CENTER

DOE - \$10,000

1977 - Continuing

**OBJECTIVES** — This environmental assessment program is analyzing and quantifying the nature of waste materials emitted into the environment by the PERC facilities and will recommend procedures for control where required.

**RECENT WORK AND ACCOMPLISHMENTS** — A room-by-room survey is currently being carried out at PERC to: identify the use of occupationally and environmentally harmful substances (types, quantities, and manner of use will be determined), estimate the quantities of the various types of pollutants released into the environment (via "product," air, wastewater, and solid wastes), identify occupationally and/or environmentally harmful practices in handling and disposal of various waste products, and evaluate and propose alternative handling and disposal techniques for the generated wastes. The information is being collected through interviews with personnel and their supervisors, reviewing past purchase orders and requisitions, materials inventory, and inspection of working practices. After analysis of these data, alternative handling and disposal methods will be proposed.

**PLANS FOR THE COMING YEAR** — Results from this study should be available by June 1978. Subsequent to the report, action will be taken on the recommendations presented. Two associated plans are being developed to deal with continuing sources and new sources. The control of continuous emissions is being carried out via a hot line from each PERC operating division to the Environment and Health Division. In the event that a spill or an inadvertent discharge to the environment occurs, it will serve as a reference point for semiannual environmental reviews.

## HEALTH PROBLEMS IN FOSSIL ENERGY DEVELOPMENT

FLOW RESOURCES CORPORATION

DOE - \$179,147

6/18/76 - 3/31/78

**OBJECTIVES** — Technical assistance is being provided to assure that possible health and safety problems associated with innovative fossil fuel technologies are identified and dealt with. Pilot plant

programs and activities were reviewed. Available literature and data were reviewed to assess the toxic, carcinogenic, or other hazardous components for which standards or data exist. Site visits were made to Energy Research Centers and Pilot Plants. The results will be used to recommend integrated approaches to occupational health. A manual workbook for health and safety matters in fossil energy plants is being prepared.

**RECENT WORK AND ACCOMPLISHMENTS** — Investigation of existing occupational health and safety activities has included acquisition of published and unpublished materials, development and distribution of a questionnaire, and site visits. Two reports have been submitted. One report concludes that existing programs tend to stress safety more than health, and to emphasize compliance and control of hazards. Consequently, most health and safety data currently available are not ideal for epidemiological surveillance. The other report describes possible methods of linking data, of follow-up for mortality studies, and of constructing work histories or exposure data. In the second phase, methods for epidemiological linkage of data were developed. The specific problems of linking morbidity indicators with work or exposure patterns were addressed. It was found that potential health problems could be categorized into three causal types: general industrial, coal related, or associated with high-temperature and high-pressure processes. Each hazard level was related to levels of program control in four main health and safety activities. From this, recommendations were developed for recordkeeping, for health and medical programs, and for industrial hygiene practices. A detailed outline for a health and safety workbook has been submitted.

**PLANS FOR THE COMING YEAR** — Project activity will be completed in 1978 with publication of the health and safety manual.

**WASTEWATER STUDY OF PERC**  
**PITTSBURGH ENERGY RESEARCH CENTER**  
**DOE - \$10,000**  
**1977 - Continuing**

**OBJECTIVES** — Variations of wastewater flow rates and pollution constituents from the PERC site will be quantified, and alternatives will be developed for sound wastewater disposal practices for PERC stormwaters based on the data collected.

**RECENT WORK AND ACCOMPLISHMENTS** — A wastewater analysis program has recently been implemented to analyze and quantify wastewater outflow from the PERC site into the main outfall pipe, which carries similar stormwater from the Bureau of Mines and Mining Enforcement and Safety Administration (MESA), into Lick Run. The entire site discharge is subject to the regulations of an NPDES permit granted by the EPA. A preliminary evaluation of PERC stormwater flows and quantitative determinations of pollution potential is necessary to assess the existence of environmental problems and to suggest viable alternatives for their resolution. The sample technique consisted of selected 5-day periods during which round-the-clock sampling was conducted. The samples collected were analyzed for key organic and inorganic pollutants by both chemical and instrumental techniques.

**PLANS FOR THE COMING YEAR** – Data obtained from the wastewater survey and analysis will be used to determine monitoring practices on site and, in part, to assess the necessity for the design and construction of an on-site treatment or disposal facility.

## **ENVIRONMENTAL SUPPORT SERVICES - SYNTHANE PILOT PLANT**

**PITTSBURGH ENERGY RESEARCH CENTER**  
DOE - \$310,000  
1977 - Continuing

**OBJECTIVES** – The Synthane pilot plant program provides support services in development, management, and review of environmental criteria applicable to Synthane pilot plant operations to insure environmentally acceptable operations. These environmental support activities incorporate five major areas: air monitoring, water monitoring, solid waste evaluation, noise assessment, and occupational health activities.

**RECENT WORK AND ACCOMPLISHMENTS** – A six-month ambient air monitoring study was begun in August 1976 as a follow-up to an earlier study. In addition, a meteorological station was established to determine the most appropriate locations for ambient air monitoring trailers. Two ambient air monitoring trailers are situated to capture airborne emissions from the Synthane pilot plant. Source sampling of the thermal oxidizer was conducted to determine compliance with air pollution regulations as well as to assist in the construction of a dispersion model for the site. As is required by the EPA, a National Pollutant Discharge Elimination System Permit was applied for and received. The permit specifies what water sources are to be sampled, how frequently they should be sampled, and what parameters are to be reported. The discharge points are the sanitary waste discharge from an extended aeration sewage treatment plant, cooling water blowdown from a once-through tower which uses potable water, demineralizer regeneration wastes from the process unit with intermittent flow, boiler blowdown high-pressure boiler, and low-pressure boiler. For further water quality assurance, continuous monitors have been set up in a new sampling pit to monitor plant in-flow and out-flow. The entire process area was underlaid with concrete to control rain runoff, spills, and maintenance wastes. Also, curbing and trenching were installed to direct the water to two large receiving tanks. These collection tanks hold the water for proper disposal. This surface water control system segregates the potentially contaminated wastes from other storm water runoff which may not contain similar levels of contamination.

Studies have been conducted on char contaminated with gasifier condensate, char processed through the filter building, and solids removed from the gasifier. Char produced from the gasification of Montana subbituminous coal was analyzed for suitability for disposal by land filling, reuse, or incineration. Noise levels recorded on tape were fed into an intermediate amplifier, graphic level recorder, and a statistical distribution recorder to obtain a representative visual record of the noise levels. The Synthane plant was chosen as the centroid for the NIOSH study "Preliminary Recommended Standards for Coal Gasification Pilot Plants." Currently, personnel monitors are being used and new requirements of dress for vessel entry are being established.

**PLANS FOR THE COMING YEAR** – The ambient air monitoring trailers and the Hi-Vols sites will continue to operate. Metals analysis will continue to change as variables within the background and plant operation are noted. Particulate size will be studied to show range and diversity. Two addi-

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tional ambient air monitoring trailers will be used on site or at other pilot plant or large-scale facilities. Occupational health work will be expanded to include in-plant monitors, Anderson impactors, Hi-Vols, and dustfall jars to characterize the nature of the contaminants in the worker environment. Non-point source sampling will also be conducted in the facility.

## **FOSSIL ENERGY ENVIRONMENTAL PROJECT**

### **OAK RIDGE NATIONAL LABORATORY**

**DOE - \$848,000**

**1977 - Continuing**

**OBJECTIVES** – Work performed under this project will provide DOE with program assistance for environmental assessment of functions related to the demonstration of advanced fossil energy conversion technologies. Assigned technical assistance tasks will be performed, and environmental investigations will be conducted that are critical to the early realization of advanced fossil energy technologies. As a vital part of fulfilling DOE's responsibilities under NEPA requirements and to aid decisionmaking processes, it is essential that site-specific impact statements be written for the major fossil energy conversion technologies under development. In addition, short-term programmatic investigations designed to assess environmental problems expected with early demonstration facilities must be carried out quickly to provide, at an early stage, the information necessary to effect rapid development of these projects.

**RECENT WORK AND ACCOMPLISHMENTS** – An environmental monitoring handbook was prepared to provide guidance to DOE's industrial partners in coal conversion demonstration programs. The handbook provides a basis for establishing viable environmental and socioeconomic monitoring programs for specific site/process situations. An introductory environmental overview, *Environmental Inclusions in Requests for Proposals*, was prepared to alert potential respondents to both initial (in the response to the RFP itself) and continuing (if they are successful bidders) environmental responsibilities. Environmental analyses of potential impacts from pipeline (high-Btu) gasification plants were carried out with a pseudo site/process-specific approach. They will provide a basis for site-specific environmental impact statements required for the first demonstration gasification plants scheduled for construction in FY 1979. Since the large quantities of solids that must be stored at coal conversion plants present potential environmental impacts, a study was undertaken to collect and assess a wide variety of relevant information. This study resulted in an environmental source book on solid-waste disposal. Definitive studies are being undertaken to determine if coal conversion wastes are to be deposited safely, economically, and permanently by landfilling. An engineering approach is being taken to assess potential impacts of leachates from stored coal and conversion wastes. Engineering data are being developed for designing landfills that will reduce groundwater impacts to acceptable levels. Environmental technical assistance was provided in a number of areas, the most significant being an advisory role to the industrial partners for the two pipeline gas demonstration plants. Specific recommendations were made on the industrial partner's proposed environmental monitoring plans. The environmental monitoring handbook, mentioned above, was used as the principal guide.

**PLANS FOR THE COMING YEAR** – The major documents initiated in FY 1977 will be finalized including the monitoring handbook, the environmental inclusions in RFPs, the environmental analysis of high-Btu gasification, and the information assessment on disposal of solid wastes. Work will begin on the EIS for the pipeline-gas demonstration plant. Technical assistance will be continued with heavy emphasis on the advisory role to industrial partners.

## DEVELOPMENT OF QUARTERLY TECHNICAL REPORTS

CAMERON ENGINEERS, INC.  
DOE - \$280,469  
6/30/76 - 4/30/78

**OBJECTIVES** – Work under this contract involves the publication of documents to summarize selected contractor activities on a quarterly and annual basis to enhance in-house control and management of contracts administered by the Division of Coal Conversion and Utilization and the Division of Major Facility Project Management. The documents also provide pertinent technical research data to other Government agencies, universities, and interested industrial organizations.

**RECENT WORK AND ACCOMPLISHMENTS** – Quarterly technical reports have been published for 76 projects covering the periods of July-December 1976 and January-September 1977. Preliminary drafts have also been prepared for the period of October-December 1977, and for the 1977 Annual Technical Report. These reports involve summaries of 15 projects in the Coal Gasification section, 21 in Power and Combustion, 18 in Coal Liquefaction, and 22 in Coal Demonstration Plants. The availability of these reports from the National Technical Information Service is listed below:

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|---|---|
| ● Coal Gasification – Quarterly Reports           | ● Coal Power and Combustion – Quarterly Reports   |
| Issued: July – Sept. 1976      NTIS: ERDA 76-93/3 | Issued: July – Sept. 1976      NTIS: ERDA 76-94/3 |
| Oct. – Dec. 1976      ERDA 76-93/4                | Oct. – Dec. 1976      ERDA 76-94/4                |
| Jan. – March 1977      DOE/ET-0024/1              | Jan. – March 1977      DOE/ET-0025/1              |
| Apr. – June 1977      DOE/ET-0024/2               | Apr. – Sept. 1977      DOE/ET-0025/3              |
| July – Sept. 1977      DOE/ET-0024/3              |   |
| ● Coal Liquefaction – Quarterly Reports           | ● Coal Demonstration Plants – Quarterly Reports   |
| Issued: July – Sept. 1976      NTIS: ERDA 76-95/3 | Issued: July – Sept. 1976      NTIS: ERDA 76-96/3 |
| Oct. – Dec. 1976      ERDA 76-95/4                | Oct. – Dec. 1976      ERDA 76-96/4                |
| Jan. – March 1977      DOE/ET-0026/1              | Jan. – March 1977      DOE/ET-0027/1              |
| Apr. – June 1977      DOE/ET-0026/2               | Apr. – June 1977      DOE/ET-0027/2               |
| July – Sept. 1977      DOE/ET-0026/3              | July – Sept. 1977      DOE/ET-0027/3              |
| ● Coal Conversion and Utilization –               | ● Coal Demonstration Plants –                     |
| 1976 Annual Technical Report      ERDA 77-86      | 1976 Annual Technical Report      ERDA 77-87      |

**PLANS FOR THE COMING YEAR** – Quarterly technical reports will be published throughout the contract period, with additional effort being devoted to the preparation of annual technical reports. As the number of DOE-sponsored projects increases, the size and scope of the technical reports will reflect this growth.

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## UNIVERSITY ACTIVITIES

Fossil Energy's University Activities program has two principal objectives: to assure the foundation for innovative technology through the use of the capabilities and talents in our academic institutions and to maintain an effective two-way channel of communication between the Department of Energy and the academic community so that trained technical manpower is available to carry out basic and applied research in support of DOE's mission.

In FY 1977, approximately 88 proposals were funded in the areas of gasification, liquefaction, materials and components, construction, oil, gas, shale, and in situ technology, and MHD at a cost of \$24 million. The unsolicited proposal system provides engineering, scientific, and baseline data that can be useful in explaining and developing processes related to Fossil Energy's goals and objectives. Twenty-eight "starter grants" (long-range exploratory research that is modestly funded) were awarded at a cost of \$940,000. Sponsorship of faculty and student participation programs has provided summer salary support for college professors, as well as students, to perform research of mutual interest at a DOE Center. DOE teacher workshops and summer institutes provide the nation's educators with up-to-date information on the energy situation and assist them with the development of educational materials for classroom use. There has been a constant interchange of information. New areas of research have been identified. New and improved technologies have been developed--all as a result of the close association maintained between the academic community and DOE.

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*Reflectance Determination — A Precise Index of Coal Rank*

## COAL LIQUEFACTION WITH APPLICATION TO SRC AND RELATED PROCESSES

AUBURN UNIVERSITY  
DOE - \$331,623; Auburn University - \$150,984  
7/1/76 - Continuing  
Principal Investigators - J.A. Guin, A.R. Tarrer

**OBJECTIVES** – The purpose of this project is to provide an increased fundamental understanding of Solvent Refined Coal (SRC) process chemistry as well as to provide guidelines and recommendations leading to economical and technical improvements in SRC technology through systematically investigating, characterizing, and delineating the effects of changes in process operating conditions, equipment configuration, and nature of raw materials upon the kinetics, mechanism, and extent of coal dissolution, heteroatom removal, and hydrogenation in the SRC and closely related processes. Specific problem areas being investigated include the effects of solvent and coal types as well as processing conditions upon coal particle dissolution; the effects of process variables, mineral matter content, and reactor configuration upon coal conversion, hydrogenation, heteroatom removal rates; and the detailed characterization of SRC recycle solvents and products by gas and liquid chromatography. These studies are valuable in selecting improved economic operating conditions and flow schemes for the SRC process, in providing an extended map of the process variable space, and in determining process kinetic models for future optimal design and scale-up to commercial-sized plants.

**RECENT WORK AND ACCOMPLISHMENTS** – An immediate impact on SRC technology is the development of a processing method by which coal mineral catalysis is used to reduce hydrogen consumption by 40 to 50 percent. In the conventional SRC process a solid boiler fuel is the main product. Some hydrogenation is required to liquefy the coal and allow the remaining solid mineral matter to be separated by physical means; however, minimum hydrogenation is desirable in order to be cost effective. In the SRC reactor, most of the coal is liquefied within a relatively short reaction time, and liquefaction asymptotically approaches only a slightly higher amount with increased reaction time. In the asymptotic region hydrogen continues to be consumed, thus it is desirable to avoid operation in this asymptotic region. Desulfurization occurs very slowly in the absence of a catalyst, and long reaction periods, extending into this asymptotic region, are required to meet new source performance standards (NSPS). Certain coal minerals like the ash of SRC residue and coal ash have been shown to selectively accelerate desulfurization over hydrogenation. Methods have been developed for using this catalytic selectivity of coal minerals to meet current requirements set by NSPS without having to operate in the asymptotic region of liquefaction. This is important in that hydrogen consumption is reduced by 40 to 50 percent.

Other accomplishments include: a detailed study of the effect and importance of initial particle size on four coals used in the SRC process; experimental data for the viscosity peaks occurring in the preheater/dissolver of the SRC process; solubility data for  $H_2$ ,  $CO_2$ , and  $CH_4$  in coal-derived oils; development of an effective model for the continuous SRC reactor; implementation of new analytical techniques, including IR, x-ray fluorescence, and Coulter counter particle size analysis; a detailed study of the variance of particle size with reaction for Kentucky No. 9/14 coal and a western, subbituminous coal; experimental data showing the effect of solvent-to-coal ratio on coal dissolution rate; and an experimental mapping of the effects of reaction time, temperature, and hydrogen partial pressure on catalytic liquefaction and desulfurization in different ranges of the process variable space.

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**PLANS FOR THE COMING YEAR** – Work will continue on coal particle dissolution studies, reaction kinetics and process variable studies, and implementation of new analytical techniques. Particular emphasis will be placed on studying the effects of hydrogen sulfide partial pressure on desulfurization; verifying and studying the inhibitive effect of CaO on liquefaction; using model compounds to evaluate the relative catalytic activity and selectivity of coal minerals; developing better analytical techniques for characterizing SRC and coal minerals; improving the accuracy of the model for the SRC reactor; and mapping studies of the process variable space.

## **POLLUTANT CONTROL THROUGH STAGED COMBUSTION OF PULVERIZED COAL**

UNIVERSITY OF ARIZONA  
DOE - \$165,052; University of Arizona - \$14,353  
4/1/75 - 3/31/78  
Principal Investigator - J.O.L. Wendt

**OBJECTIVES** – This project focuses on the development of new pulverized coal combustion technology with a view to minimize the amount of pollutants emitted. Emphasis is on nitrogen oxide emissions, a significant portion of which can be attributable to oxidation of chemically bound nitrogen in the fuel. Staged combustion, in which a portion of the combustion air is removed from the normal burner air and added at some distance downstream, appears to be an effective combustion modification; yet the maximum extent that “Fuel NO<sub>x</sub>” can be controlled by this method is unknown. The two specific objectives addressed in this contract are (1) to determine quantitatively those factors that influence NO<sub>x</sub> emissions from pulverized coal combustion and (2) to define the optimum staged combustion configuration for low NO<sub>x</sub> emissions. Success in this research will, therefore, provide technical guidelines that allow increased utilization of coal through combustion without excessive environmental damage.

**RECENT WORK AND ACCOMPLISHMENTS** – The first objective has been met, and results have been reported elsewhere. The experimental combustor has been modified to allow a plane pulverized coal flame to be established and to allow time resolved species and temperature profiles to be obtained under both fuel rich and staged combustion conditions. The salient features of fuel nitrogen conversion mechanisms have been determined. It was found that the major limitations to increased NO<sub>x</sub> abatement through classical staging arise from (1) a high initial rate of NO formation which occurs even under very fuel-rich conditions; (2) a slow rate of NO reduction subsequent to this; and (3) the formation of “second stage” NO resulting from the carryover of nitrogenous species from the first stage. The practical significance of these limitations is that very low NO levels can be achieved only by holding the first stage very fuel rich for very long periods of time and even then, only with difficulty. As a result, a new staging concept was devised. This modification, denoted as Advanced Staging, segments the combustion into three or more, rather than two, zones maintaining very fuel rich conditions for only very short times, i.e., less than 0.3 sec. This method enabled exhaust values of 100 ppm to be obtained which is equivalent to 90 percent abatement of NO<sub>x</sub>.

**PLANS FOR THE COMING YEAR** – Results will be extended to five different coals and several coal chars. The Advanced Staging concept will be pursued, and attempts to determine the optimum configuration will be made.



## HIGH-PRESSURE, STIRRED, NEAR-PLUG FLOW LABORATORY REACTOR

UNIVERSITY OF ARIZONA  
DOE - \$20,000; University of Arizona - \$1084  
8/15/77 - 8/14/78  
Principal Investigator - D.H. White

**OBJECTIVES** — This project intends to prove the feasibility, the advantages, and the limitations of a proposed new laboratory reactor for handling coal slurries in coal liquefaction and related research. An existing polymer plasticating extruder is to be modified so that it will act as a high-pressure, stirred, near-plug flow laboratory reactor from which dense coal slurry samples can be taken.

**RECENT WORK AND ACCOMPLISHMENTS** — The project is in its initial stages, wherein a closed feed system and certain sampling devices are being added to the extruder reactor. Initial trial runs are utilizing a pulverized coal/fuel oil slurry.

**PLANS FOR THE COMING YEAR** — After necessary extruder reactor modifications have been made, operating data on coal slurries simulating coal liquefaction will be obtained to determine reactor feasibility, ease of operation, and advantages/limitations of the system. Auxiliary operating procedures on sampling techniques, residence time distribution (RTD), fast heating to reaction temperatures, and fast quenching of products will be developed. If results are encouraging, then a preliminary design of a new laboratory reactor (based on findings of this project) will be made.

## COAL GASIFICATION AND COMBUSTION IN MOLTEN MEDIA

CALIFORNIA INSTITUTE OF TECHNOLOGY  
DOE - \$22,500  
9/1/76 - Continuing  
Principal Investigator - G.R. Gavalas

**OBJECTIVES** — This project seeks to investigate fundamental aspects of coal gasification in molten carbonates and phosphates. This process can be used for both high- and low-Btu gas or synthesis gas and has certain attractive features, including convenient sulfur removal and the possibility of separate evolution of the products of steam-carbon and oxygen-carbon reactions. Specific objectives are to investigate the mode of catalytic actions; i.e., whether the steam-char and oxygen-char reactions occur directly or through an intermediate species in the melt. The dependence of gasification rates on fluid mechanical variables, such as bubble size and mixing patterns, will also be studied. Another objective is to study the suitability of phosphate melts as compared to carbonate melts.

**RECENT WORK AND ACCOMPLISHMENTS** — An extensive survey was made to identify molten salts that meet the following requirements: (1) sufficiently low melting point, (2) low volatility, (3) stability at high temperatures in the presence of  $O_2$ ,  $H_2O$ ,  $CO$ ,  $CO_2$ , and (4) low cost and low toxicity. In addition to sodium carbonate, which is used in the Kellogg and the Atomics International processes, sodium phosphate,  $xNa_2O + yP_2O_5$  was found quite attractive. This non-stoichiometric compound has a low melting point forming an eutectic of  $580^\circ C$  at composition of 43 percent  $P_2O_5$  molar. The phosphate melt was found to be nonvolatile and to hydrolyze to a very low extent. Preliminary studies of steam-char reaction in molten sodium phosphate-silicate were

performed in a bench-scale system consisting of an alumina crucible enclosed in a furnace. The reaction products were analyzed in a continuous IR detector. It was found that phosphate and carbonate melts have about the same activity, e.g., at 1050°C the reaction rate observed was 8 gC/gC in melt/hr. The reaction rate was found to decrease as the SiO<sub>2</sub> content of the melt increased; for example when the content of SiO<sub>2</sub> was 20 percent the reaction rate at 1050°C decreased by a factor of three. A cold model was constructed to study the movement and coalescence of bubbles in solid-liquid suspension similar to the char-melt suspension which cannot be observed directly.

**PLANS FOR THE COMING YEAR** – In the coming year, plans are to construct a new reactor system that will allow better mixing patterns and higher steam pressure. The steam-char reaction kinetics in melts of various compositions will be studied to determine the reaction mechanism and the effect of fluid mechanical variables. The main mechanistic problem is whether the reaction is by direct steam-char contact or is mediated by a species in the melt. Such species might include sulfates formed by the sulfur in the coal and hydroxyl groups attached on the phosphate chains. The study of fluid mechanical variables will include the movement and coalescence of steam bubbles in the melt, including the effect of the wetting angle between char and melt. Later studies will include the oxygen-char reaction and the purification of the phosphate melt from accumulating coal ash impurities.

## DEW POINTS IN HOT, TAR-CONTAINING GASES

UNIVERSITY OF CALIFORNIA, BERKELEY

DOE - \$119,542; UCB - \$7360

6/1/76 - 5/30/79

Principal Investigator - J.M. Prausnitz

**OBJECTIVES** – The purpose of this research is to establish an engineering-oriented molecular-thermodynamic framework for predicting dew-point temperatures for tar-containing gases at high pressure and temperature, coming from coal gasifiers. Since these hot gases must be cooled in heat exchangers for sensible-heat recovery, it is important to prevent condensation of tars lest the heat exchanger be plugged by condensate.

**RECENT WORK AND ACCOMPLISHMENTS** – To obtain fundamental physico-parameters, two experimental studies are in progress. The first experimental study measures vapor pressures of high-boiling hydrocarbons (completed) and hydrocarbon derivatives (in progress). The second experimental study measures solubilities of liquid (or solid) hydrocarbons (hydrocarbon derivatives) in compressed gases such as methane. Measurements have been made for a variety of hydrocarbons in the region 25-275°C. The apparatus is now undergoing modification to permit measurements at higher temperatures. Mathematical molecular models have been developed to calculate those thermodynamic properties that are needed to calculate dew-point temperatures. While present work has been confined to pure liquids, extension to tars is now underway. To characterize tars, a spinning-band distillation column is used. This column separates an industrially obtained tar into (approximately) 10 fractions, each having a different boiling point.

**PLANS FOR THE COMING YEAR** – Additional experimental data will be obtained for representative hydrocarbon liquids (and solids) and representative gases, primarily methane and hydrogen. Then, experimental data will be obtained for a variety of tar fractions. The molecular models will be

tested and modified as needed. Computer programs will be developed for reducing the molecular-thermodynamic models to engineering design practice.

### LOW ALLOY STEELS FOR THICK WALL PRESSURE VESSELS

LAWRENCE BERKELEY LABORATORY, UNIVERSITY OF CALIFORNIA

DOE - \$160,000

10/1/76 - Continuing

Principal Investigators - E.R. Parker, V.F. Zackay

**OBJECTIVES** – This project seeks to adapt or develop low alloy steels that can be field fabricated into large-diameter thick-wall pressure vessels for coal gasification systems. The work centers about developmental studies on commercially available steels and on new alloy steel systems to meet this goal. The successful completion of this alloy development program will result in better steels for thick section pressure vessels. These steels will have sufficient fabrication “forgiveness” to allow on-site construction of containment vessels requiring limited post-weld heat treatments. The steels will have improved environmental resistance and will have adequate strength and toughness in thick section to ensure reliable operation of large reaction vessels.

**RECENT WORK AND ACCOMPLISHMENTS** – Research during the first year has been directed toward five program tasks: (1) determining alloy design criteria; (2) developing methods of simulating thick plate material in the laboratory; (3) characterizing commercial steels in thick sections; (4) modifying existing steels to achieve desired properties; and (5) developing new steels better suited to thick wall application. The first three tasks have been completed successfully and have been used as the basis for developing modified commercial steels with better properties. Two alloy steel systems, Mn-Mo-Ni steels and Cr-Mo steels, have served as the basis for the research. The Mn-Mo-Ni steels have been alloyed for improved strength, toughness, and environmental resistance. Three new alloys have been selected for thorough property investigation. The Cr-Mo steels have been alloyed for increased hardenability. Uniform thick section microstructures have been achieved leading to improved strength and toughness.

**PLANS FOR THE COMING YEAR** – Thorough characterizations of promising modified commercial steels will be undertaken. This characterization will entail mechanical property assessment in different thicknesses and heat treatments. Environmental resistance and weldability will also be evaluated. New steels, relying on eutectoid decomposition for strength, will also be evaluated.

### WEAR RESISTANT ALLOYS FOR COAL HANDLING EQUIPMENT

LAWRENCE BERKELEY LABORATORY, UNIVERSITY OF CALIFORNIA

DOE - \$225,000

1/1/77 - Continuing

Principal Investigators - E.R. Parker, V.F. Zackay

**OBJECTIVES** – This program intends to develop wear resistant alloys for coal transportation and fragmentation equipment. It is anticipated that large tonnages of coal will be mined, sized, transported, and handled because of the move towards utilizing fossil fuels for energy production. Coal contains abrasive particles and the handling of large tonnages will cause severe wear problems in equipment. There is, therefore, a need to develop better abrasion resistant alloys, and this program is geared towards achieving that goal.

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**RECENT WORK AND ACCOMPLISHMENTS** – The inservice operating conditions of coal transport and fragmentation equipment were identified. It was concluded that various combinations of dry or liquid slurry abrasion, impact loading, and temperatures varying from ambient up to 1100°F can be encountered. A number of laboratory abrasion tests were identified as possible candidates for use as simulators of actual service conditions and for providing wear data for ranking of experimental alloys. A dry abrasive wear tester was also designed, constructed, and calibrated. A number of steels were investigated, and work to date indicates that the low-alloy ultra-high-strength steels could be suitable for ambient temperature applications, and secondary hardening steels would be useful for applications involving elevated temperatures.

**PLANS FOR THE COMING YEAR** – The abrasive wear properties of the developmental alloys will be determined using different testers based on the application for which the steel is being considered. The metallurgical characterization of the experimental steels will be completed. A wear tester to simulate three-body wear at ambient and elevated temperatures will be constructed and used for ranking of the experimental steels.

### **OXYGEN STOICHIOMETRY AND ANALYSIS OF COAL, LIGNITE, COKE, AND THEIR DERIVATIVES**

UNIVERSITY OF CALIFORNIA, IRVINE

DOE - \$96,136; UCI - \$6400

3/1/76 - 3/1/78

Principal Investigators - G.E. Miller, A. Volborth

**OBJECTIVES** – This program is to fully examine the contribution that neutron activation techniques make to elemental analysis of fossil fuel materials. Successful development would lead to recommendations for routine methods to supplement those currently used in support of mining and processing operations.

**RECENT WORK AND ACCOMPLISHMENTS** – Several hundred samples representing over 100 coals have been analyzed for oxygen using the  $^{16}\text{O}(\text{n,p})^{16}\text{N}$  reaction with 14 MeV neutrons. Many of these samples have been carefully prepared by several different drying techniques (vacuum, oven, "Brabender") in order to examine the complex relationship between the chemical nature of the raw materials and their behavior on heating. Since such methods are utilized in standard sample preparation for classical analyses, it is important to establish the mechanism by which errors may be introduced. Analyses of related ashes and volatiles have also been performed to complete the information base to enable a better understanding of the stoichiometry of the system. Present efforts are directed to the correlation and interpretation of the data collected in terms of the type and rank of raw coal, moisture, ash, and energy contents. Sufficient correlation may exist that oxygen determination could be employed as a routine diagnostic tool to give preliminary information on new materials. A considerable number of samples have also been analyzed, using similar neutron activation methods, for nitrogen and silicon. This method also shows promise for development as a routine technique. Current work involves establishing optimum conditions for these measurements and extending the determination to include other elements simultaneously.

**PLANS FOR THE COMING YEAR** – Efforts in data correlation will continue as well as further optimization of the experimental system. This work is being carried out in cooperation with North Dakota State University to obtain comparison data on a system established for and dedicated to fossil energy work as final evidence of the viability of such methods in routine analysis.

## CORROSION OF STRUCTURAL MATERIALS IN CONTACT WITH COAL CHARs

UNIVERSITY OF CALIFORNIA AT LOS ANGELES

DOE - \$123,574; UCLA - \$11,519

3/1/77 - 2/28/80

Principal Investigator - D.L. Douglass

**OBJECTIVES** — It is planned to determine the extent of reaction of six superalloys exposed to coal chars at elevated temperatures (1600° to 1800°F) for long periods of time. Both low- and high-sulfur chars will be used. The mechanism(s) of the reaction and the principal modes by which degradation of the metals occurs will be determined. It is also planned to determine the transport rate of carbon through various oxides that form as reaction products in coal gasifiers. Ultimately the selection of alloys for structural components in coal gasifiers will be made so that the anticipated lifetime of the components can be attained in actual use.

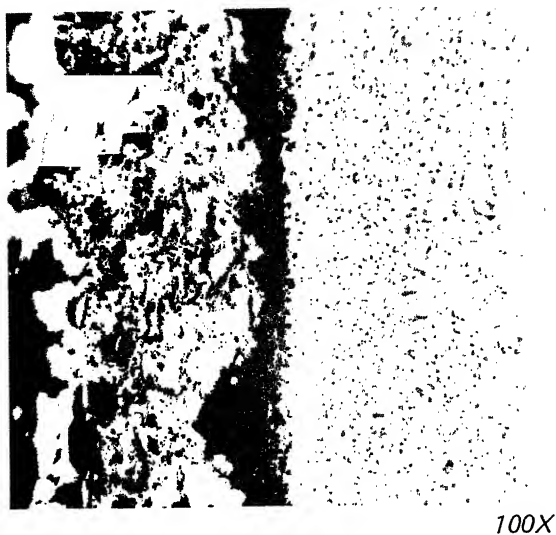
**RECENT WORK AND ACCOMPLISHMENTS** — Three nickel-base superalloys, type 310 stainless steel, and one cobalt-base superalloy have been obtained. The nickel-base alloys and the stainless steel have been exposed for preliminary runs at 1800°F for times up to 50 hours in both chars with an argon atmosphere. The corrosion rates were higher in the high-sulfur (2.7 percent) char than in the low-sulfur (0.9 percent) char, the greatest difference existing for the 310 stainless steel. Data for samples exposed 50 hours at 1800°F are listed below. Metallographic analysis of mounted samples in cross section revealed extensive grain boundary attack beneath the complex sulfide films formed on the surface. X-ray dispersive energy analysis showed silicon, sulfur, chromium, and iron in the grain-boundary reaction product. However, carbon cannot be detected with the existing instrument, and it is not presently known if the grain-boundary phase is a sulfide or carbide.

**WEIGHT GAIN DATA FOR VARIOUS ALLOYS EMBEDDED  
IN CHAR FOR 50 HOURS AT 1800°F**

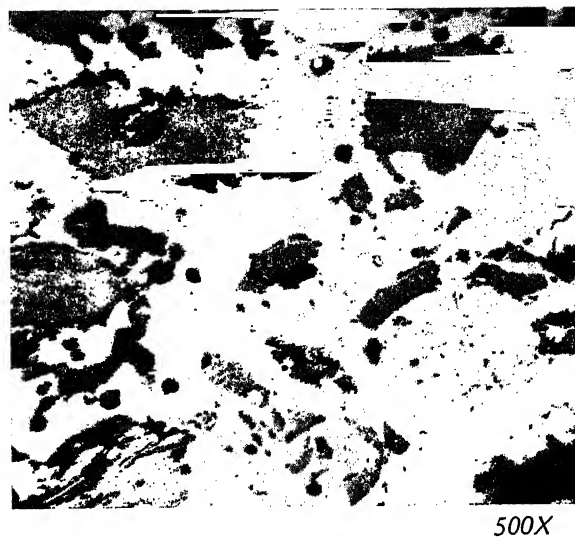
| Alloy         | Weight Gain, mg/cm <sup>2</sup> |                            |
|---------------|---------------------------------|----------------------------|
|               | FMC Char (2.7 percent S)        | Husky Char (0.9 percent S) |
| 310 Stainless | 20.55                           | 8.09                       |
| Hastelloy-X   | 16.24                           | 9.28                       |
| Inconel 671   | 13.09                           | 8.58                       |
| Incoloy 800   | 10.42                           | 8.60                       |

The preliminary studies were performed in an argon atmosphere instead of in an actual coal-gasifier atmosphere for reasons of convenience and simplicity. In order to establish if this atmosphere is more active or less active than the actual ones, runs were made in the high-sulfur char in the MPC/IITRI atmosphere\* by Mr. R.A. Perkins, Lockheed Research Laboratory. The samples (three nickel-base alloys and 310 stainless) were immersed in a refractory boat of char so that only 1/8 of the sample protruded above the char level. Hastelloy-X and In-800 exhibited severe slagging (forma-

\*36.6 percent H<sub>2</sub>, 30.5 percent H<sub>2</sub>, 20.6 percent CO, 10.9 percent CO<sub>2</sub>, 0.88 percent H<sub>2</sub>S (P<sub>O<sub>2</sub></sub> = 9.9 x 10<sup>-6</sup> atm., P<sub>S<sub>2</sub></sub> = 2.4 x 10<sup>-6</sup> atm).



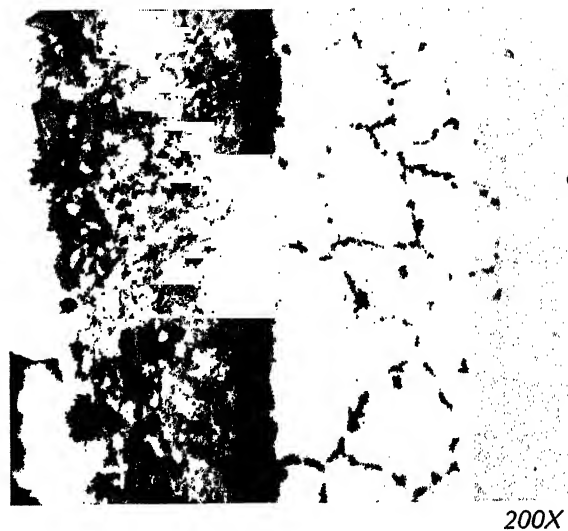
(a) Hastelloy X



(b) Hastelloy X



(c) 310 Stainless Steel



(d) Incoloy 800

*Microstructure of Alloys Exposed to High-Sulfur Char for 50 Hours at 1800°F*

tion of molten sulphides) and extensive reaction. Inconel 671 and 310 stainless appeared resistant to the char and atmosphere for the duration of the test, 96 hours.

**PLANS FOR THE COMING YEAR** – Evaluation of the atmosphere over the char will be made with respect to differences noted in corrosion rates. If the MPC/IITRI atmosphere is more severe, it will be used exclusively. Once the atmosphere has been selected, long-time runs of several hundred hours will be made so that the reaction kinetics can be determined. The temperature dependence of the rate will be measured as well. The reaction products will be characterized by metallography,

x-ray diffraction, and dispersive energy analysis in order to identify all phases and proposed reaction mechanisms. Screening tests of various experimental Ni-Cr-Al alloys which form  $\text{Al}_2\text{O}_3$  films during oxidation will be completed, and a commercial superalloy that is the closest in composition to the best screening alloys will be obtained and extensively studied. Carbon diffusivities in  $\text{Cr}_2\text{O}_3$  will be measured using Auger spectroscopy and ion sputter etching. Simultaneously, experiments will continue in which pregrown films will be exposed to carbonaceous atmospheres. The time required to form carbide layers and/or internal carbides will be used to calculate diffusivities when the thickness of the pregrown film is known.

## CHEMISTRY AND STRUCTURE OF COAL-DERIVED ASPHALTENES

UNIVERSITY OF SOUTHERN CALIFORNIA  
DOE - \$91,465; University of Southern California - \$2323  
7/1/75 - 6/30/78  
Principal Investigator - T.F. Yen

**OBJECTIVES** – This project is studying the basic science of asphaltene in order to elucidate its role in coal liquids. Understanding the chemistry and structure of coal-derived asphaltene is essential to the development of an efficient coal liquefaction process. Asphaltene affects the amount and quality of oil produced. Knowledge regarding the generation of asphaltene will help to eliminate coke formation. Heterocyclic atoms (sulfur, nitrogen, oxygen) as well as metals and ashes tend to concentrate in the asphaltene fraction. Asphaltene and its high molecular weight derivatives (carbene and carboid) are responsible for high viscosity, solvent incompatibility, and processing instabilities. It is anticipated that correlation of structural parameters with conversion and refining variables will yield useful information for the design and operation of an efficient coal liquefaction process.

**RECENT WORK AND ACCOMPLISHMENTS** – A reproducible method has been developed for the separation of any coal liquid into the following five solvent fractions: oil, resin, asphaltene, carbene, and carboid. These fractions cumulatively account for over 95 percent of any particular coal liquid investigated. Asphaltene can then be further separated by solvent elution chromatography with benzene, diethyl ether, and tetrahydrofuran, on silica gel. Three distinct fractions may be obtained with 98 to 99 percent recovery of starting asphaltene. These three fractions have been characterized by a variety of analytical, physical, and chemical methods. The diethyl ether eluted fraction is found to be more basic and polar, to have a higher degree of saturation, and to contain smaller average aromatic  $\pi$ -systems than the benzene eluted asphaltene fraction. The third fraction (THF-eluted) has the highest molecular weights and the largest percentages of nitrogen and oxygen. It is believed to be solubilized originally in benzene by association with the other two fractions. Information has been obtained that is useful for correlation with the conversion variables. At present, it is possible to conjecture the following. (1) Coal liquids are in dynamic equilibrium; asphaltene can be converted to carbene, then to carboid. Asphaltene also can be converted to resins and then to oil. The controlling factors seem to be conversion variables such as temperature, pressure, solvent, catalyst and space velocity. (2) Preheating in any coal conversion process merely converts coal to primarily asphaltene and carbene-carboid fractions. The major function of catalytic reactor is to catalytically hydrogenate the asphaltene and carbene-carboid fractions, which is an easier process than hydrogenating the coal directly. Proper combination and configuration of a preheater and catalytic reactor will lead to a more efficient process. Coal-derived asphaltene is a smaller and less complex system than petroleum-derived asphaltene; therefore, it is anticipated that coal asphaltene may be refined or upgraded with ease.

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**PLANS FOR THE COMING YEAR** — Characterization of asphaltene and its derivatives will be continued by electron spin resonance, polarography, gel permeation chromatography, oxidative degradation studies, and chemical functional group determination by silylation and methylation.

## APPLICATIONS OF DIELS-ALDER REACTION TO COAL FRACTIONATIONS

UNIVERSITY OF SOUTHERN CALIFORNIA

DOE - \$40,000

8/1/77 - 7/31/79

Principal Investigator - T.F. Yen

**OBJECTIVES** — The technical goals of this research are twofold: first, to gain knowledge of the basic science as to how the different coal components are held together, thus providing valuable insights into the process of taking them apart; and secondly to examine the feasibility of applying the Diels-Alder reaction to fractionating coal into useful raw materials. The practical advantages of this proposed work will be: comminution or size reduction of coal; separation of pyrite or other minerals; and fractionation of coal (at least the olefins).

**RECENT WORK AND ACCOMPLISHMENTS** — Three coal samples—low-volatile bituminous, high-volatile bituminous, and lignite from Penn State—have been subjected to a study of solubility parameter spectra. The solvent ranges are from an effective solubility parameter of 6 to 23 hildebrands. A number of different coals were subjected to a number of coal extraction schemes for the isolation of different fractions.

**PLANS FOR THE COMING YEAR** — Diels-Alder type of dienes and dienophiles will be used as coal extraction solvents.

## COAL PARTICLE COMBUSTION

STANFORD UNIVERSITY

DOE - \$150,000

7/1/77 - 6/30/79

Principal Investigator - C.H. Kruger

**OBJECTIVES** — This research project seeks to experimentally determine mechanisms and rates of pulverized coal particle combustion. Specific objectives of this research are: (1) development of an integrated high-temperature flow reactor and measurement system which is designed to give maximum accuracy in the experimentally determined pulverized coal reaction rates; (2) application of the flow reactor system to determine reaction rates for representative coals burned under fuel rich conditions ranging from 50 percent to 100 percent stoichiometric air; (3) investigation of physical effects, including ash melting and particle breakup on the primary mechanism of combustion and the resulting effect on coal reaction rates. The results of these investigations will be presented in a practical and useable form for potential design users.

**RECENT WORK AND ACCOMPLISHMENTS** — Preliminary design of the high-temperature flow reactor has been completed. Two interchangeable reactor tubes (4-inch and 5-inch diameter) coupled with the 5 to 45 gm/sec flowrate range of the arc jet allow investigation of reaction times varying from 20 to 300 msec. These residence times will cover the range of reaction times for pulverized coal ( $\leq 150 \mu\text{m}$  diameter) burning in an oxidizing atmosphere in the temperature range



1500° to 2000°K. The primary features of the reactor design are summarized as follows: (1) Uniform flow is obtained in the central core region of the two reactor tubes which allows for accurate centerline probing as well as line-of-sight optical measurements at the reactor exit; (2) Axial temperature drops are  $\leq 3$  percent in the worst case, indicating that the reactor will approach ideal isothermal reaction conditions; (3) Coal particle loss from the main flow via deposition on the reactor walls is  $\leq 20$  percent for all flow conditions; (4) The plenum, flow straightener, contraction cone, and diffuser sections are designed to both mix the reactants and provide well-characterized flow conditions at the entry of the flow reactor. Knowledge of the flow conditions will allow corrections for the small non-ideal effects mentioned in items 1, 2, and 3.

**PLANS FOR THE COMING YEAR** – Final design of the system, hardware specification, and ordering is proceeding. While hardware fabrication is occurring, diagnostic equipment will be assembled and tested. Development of a complete data reduction algorithm will also proceed. System components will be assembled in spring 1978, and initial checkout and preliminary testing of the system will begin by the summer. The remaining part of the year will focus on obtaining preliminary measurements of the dependence of reaction times on gas phase temperature, fuel-oxidizer stoichiometry, coal type, and size distribution.

### CLEAN SOLID AND LIQUID FUELS FROM COAL

COLORADO SCHOOL OF MINES  
DOE - \$570,967; State of Colorado - \$82,921  
10/1/75 - 10/1/78  
Principal Investigators - J.H. Gary, J.O. Golden

**OBJECTIVES** – This contract investigates processing of coal to clean solid and/or liquid fuels. Research is being carried out in five different areas: (1) Kinetics and mechanism of coal hydrodesulfurization and liquefaction during dissolution in a bath reactor; (2) development of a disposable shift reaction catalyst for the CO-Steam coal liquefaction system; (3) screening of conventional petroleum catalysts for hydrodenitrogenation of a coal-derived liquid; (4) reactor configuration and kinetic studies for coal liquefaction/hydrodesulfurization in a continuous-flow pilot plant; (5) analytical studies aimed at improving techniques for analysis of coal liquefaction products and heteroatom content of reactants and products.

**RECENT WORK AND ACCOMPLISHMENTS** – During the past year, studies on desulfurization and liquefaction kinetics have been completed. Rates of organic sulfur removal during hydrogenation in solution and formation of gross liquid components (preasphaltenes, asphaltenes, oils) using a donor solvent have been investigated. Application of disposable shift reaction/hydrogenation catalysts to the CO-Steam coal liquefaction system is being studied. Preliminary screening of several catalysts is in progress, with results to date indicating pronounced increases in gas-phase hydrogen due to the presence of certain catalyst systems. Screening of conventional petroleum HDN and HDS catalysts for use on coal-derived liquids is underway. Catalysts have been studied in batch stirred, continuous stirred, and trickle-bed reactors to obtain initial activity and catalyst life data. Reactor studies (stirred tank/plug flow) in continuous flow equipment are now in progress. A bench-scale unit allowing comparative kinetic data to be obtained for both reactor configurations is in operation and will be utilized for obtaining steady-flow rate data. Goals are to develop realistic reactor models for both stirred tank and plug flow as functions of reactor operating parameters (i.e., temperature, pressure, solvent-to-coal ratio, hydrodynamics, residence time). Particular attention is being paid to

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the fate of heteroatom compounds and gross hydrocarbon classifications in the studies. Analytical studies have resulted in an improved procedure for analysis of total sulfur and sulfur forms in coal and coal-derived liquids. Specific techniques for isolating nitrogen-rich fractions from coal liquids have also been developed, as have improved instrumental procedures for rapid screening of heteroatom content in parent coal and liquefaction products.

**PLANS FOR THE COMING YEAR** – Continuation of the work on kinetics and mechanisms will center on model discrimination, on mechanistic studies, and on more fundamental investigations of the fate of functional groups and organic compounds during hydrogenation. This research will be carried out using batch reactors that can be quick-charged with reactants and quenched to minimize effects due to heat-up and cool-down. Future work on disposable CO-Steam catalyst systems will focus on comparison of catalytic CO-Steam kinetics for organic sulfur removal with non-catalytic desulfurization mentioned previously. The HDN catalyst evaluation program is being continued for long-term testing of a group of selected catalysts with varying metal contents, support compositions, and pore size distributions to obtain steady-state data. Work on the bench-scale continuous processing unit will continue with both stirred tank and plug flow data taken at equivalent residence times. Continued work on analytical improvements will center on instrumental analysis techniques (Mössbauer spectroscopy, HPLC) and on development of rapid separation schemes that are specific for liquids analysis.

## ENTHALPY MEASUREMENT OF COAL-DERIVED LIQUIDS

COLORADO SCHOOL OF MINES  
DOE - \$277,683; Colorado School of Mines - \$30,584  
6/24/75 - 6/23/78  
Principal Investigators - A.J. Kidnay, V.F. Yesavage

**OBJECTIVES** – Thermodynamic property research is recognized as invaluable by process and design engineers in the petroleum, chemical, and allied industries. Calorimetric measurements of specific heats or enthalpies, pressure-density-temperature measurements, and phase equilibrium determinations, for pure fluids or complex mixtures, are all essential for the optimum design of both physical and chemical processing units. The research is divided into three major program areas: (1) Design, construction, and evaluation of freon boil-off calorimeter for temperatures of 70° to 700°F and pressures to 2000 psig; (2) enthalpy measurements on approximately 10 samples of coal-derived liquids; (3) preparation of engineering correlations for the measured enthalpy data and comparison with representative data for petroleum and petroleum fractions.

**RECENT WORK AND ACCOMPLISHMENTS** – During the past year, the evaluation of the calorimetric facility was completed. Data were obtained for water over the range 65° to 551°F, 179 to 1529 psia and with enthalpy differences from 130 to 511 Btu/lb<sub>m</sub>. Data were also obtained for n-Heptane over the liquid, two phase, and vapor regions. Based on these data an overall accuracy of ± 0.5 percent was demonstrated for the calorimeter. This compares very well with other similar calorimeter facilities. Measurements were also made on a number of samples of coal-derived liquids. The correlation of enthalpies for coal-derived liquids has as its logical starting point the correlations already in existence for petroleum fractions. During the year, several of the most popular methods of enthalpy prediction developed for petroleum fractions were compared with experimental enthalpies of coal-derived liquids. At lower temperatures (≈300°F) predicted enthalpies compared well with experimental enthalpies, but at higher temperatures (≈700°F) the predicted values were substantially in error.

**PLANS FOR THE COMING YEAR** – During the coming year work will continue on both the enthalpy measurements of coal-derived liquids and on the evaluation of existing enthalpy correlations. In addition, work will be initiated on the enthalpy measurements of “model compounds” (i.e., materials known or believed to be major constituents of coal-derived liquids) and on the development of new correlations for enthalpy.

#### COAL-DERIVED LIQUID SAMPLES

| Coal                | Conversion Process | Organization Furnishing Sample           | Number of Enthalpy Measurements | Temperature Range °F | Pressure Range psia | Comments                      |
|---------------------|--------------------|--|---------------------------------|----------------------|---------------------|-------------------------------|
| Western Kentucky    | COED               | Bartlesville Energy Research Center      | 66                              | 65-750               | 100-1500            | Whole oil sample as furnished |
| Utah                | COED               | Bartlesville Energy Research Center      | 1                               | 345                  | 627                 | Whole oil sample as furnished |
| Western Kentucky    | COED               | Bartlesville Energy Research Center      | 81                              | 65-750               | 60-500              | Distillate, 650°F endpoint    |
| Utah                | COED               | Bartlesville Energy Research Center      | 86                              | 65-720               | 60-1500             | Distillate, 520°F endpoint    |
| Kentucky Bituminous | SRC-I              | Pittsburgh Energy Research Center        | 57                              | 155-740              | 150-1500            | Distillate, 520°F endpoint    |
| Kentucky Bituminous | SRC-I              | Pittsburg and Midway Coal Mining Company | Measurements in progress        |                      |                     | Naptha                        |

#### A NEW PROCESS FOR PRODUCING CARBON MONOXIDE AND HYDROGEN

COLORADO SCHOOL OF MINES  
DOE - \$40,000; Colorado School of Mines - \$7188  
9/77 - 9/79  
Principal Investigator - E.D. Sloan

**OBJECTIVES** – Basic kinetic data are provided on the reaction  $P_4O_{10} + 10 C \rightarrow 10CO + P_4$  as a second reaction in a coal gasification process. A secondary objective is to consider  $P_4O_{10}$  solid phase transitions. The combination of the first reaction with a second better known reaction  $P_4 + 10H_2O \rightarrow P_4O_{10} + H_2$  yields two combustible gases from C and  $H_2O$ . Basic kinetic data on the initial reaction will lead to pilot plant studies in future work.

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**RECENT WORK AND ACCOMPLISHMENTS** – During the initial months of work, thermodynamic calculations have been made to determine all possible reactants and product concentrations in the first reaction. Means of accurate product analysis from the reaction are currently being considered.

**PLANS FOR THE COMING YEAR** – The gas analysis system will be built and initial kinetic studies will be performed in a tubular reactor. Improved reactor designs will be made for better kinetic data.

#### EXPERIMENTAL VERIFICATION OF A NEW COAL LIQUEFACTION TECHNIQUE

COLORADO SCHOOL OF MINES  
DOE - \$20,000  
9/1/77 - 8/31/78  
Principal Investigator - M.S. Graboski

**OBJECTIVES** – This proposal intends to test a new concept that utilizes intensive microwave heating and a donor solvent to liquefy coal at atmospheric pressure using short residence times. A second objective is to briefly study the effect of the major variables on the process yield so that a preliminary judgement on ultimate potential of the process can be made. Coal liquefaction systems under investigation require high temperatures, hydrogen partial pressures, and long residence times to produce high yields of liquid products. This process potentially can reduce the severe conditions required to liquefy coal, thus greatly simplifying the plant design. The process concept should also minimize secondary decomposition of liquids to gases and tars.

**RECENT WORK AND ACCOMPLISHMENTS** – The experimental system has been designed and the equipment orders are being placed. Mathematical modeling of the process is being carried out to estimate the effect of variables, such as coal particle size on the process performance.

**PLANS FOR THE COMING YEAR** – Laboratory experiments will be performed to determine the effect of the major variables on process yield. A 9-month performance period is anticipated, allowing 6 months to design, procure, and assemble the bench-scale apparatus, and 3 months to conduct the basic experiments. At that point a preliminary judgement on the process potential can be made.

#### DEVELOPMENT OF SULFUR TOLERANT METHANATION CATALYSTS

UNIVERSITY OF COLORADO  
DOE - \$40,000; University of Colorado - \$5100  
9/77 - 8/79  
Principal Investigator - M.C. Rakowski

**OBJECTIVES** – The objective of the proposed research is to develop effective methanation catalysts for use in single-stage processes that gasify and convert coal to synthetic natural gas in a single reactor. The external energy required for the endothermic coal gasification process is significantly reduced in such a system because of the available heat produced by the exothermic methanation reaction. However, the nickel catalysts presently used in the single-stage reactors are poisoned by the sulfur compounds which are released into the reactor by the coal gasification process. The proposed program will develop catalytic systems for the methanation reaction that are

not subject to sulfur poisoning. These catalysts would increase the efficiency of the energy-conserving single stage coal reactors.

**RECENT WORK AND ACCOMPLISHMENTS** — Chemicals and equipment required for the study have been purchased. The following molybdenum complexes, which are proposed as potential catalysts or catalyst precursors, have been synthesized:  $C_5H_5Mo(CO)_3H$ ,  $[C_5H_5Mo(CH_3S)_2]_2$ , and  $[(C_2H_5)_2NCS_2]_2MoOS_2$ . The preparation of additional complexes is in progress. Standard conditions for catalytic reactions are being established. Chromatographic conditions which will permit ready identification of hydrogenation products are currently being developed.

**PLANS FOR THE COMING YEAR** — The synthesis of a series of molybdenum sulfur complexes will be completed and their catalytic activity will be determined. Variations of catalyst surface area, reaction temperatures, and gas pressures will be studied in order to determine their effect on catalyst activity. Catalyst efficiency in the presence of hydrogen sulfide and other sulfur "poisons" will be determined. Studies will be initiated to explore the possibility of promoting catalytic activity by chemically pretreating the original complexes.

#### SIMULATED NONEQUILIBRIUM ENERGY DISTRIBUTIONS IN STEAM/COAL CHAR REACTION

UNIVERSITY OF COLORADO  
DOE - \$47,346; University of Colorado - \$1819  
3/1/76 - 2/28/78  
Principal Investigator - L.F. Brown

**OBJECTIVES** — This work is determining the feasibility of inducing nonequilibrium molecular energy distributions in a reacting coal char/steam system for the purpose of allowing the reaction to be run at a substantially reduced temperature.

**RECENT WORK AND ACCOMPLISHMENTS** — The project has been divided into four phases: I, study of gas-phase excitation; II, study of solid-phase excitation; III, determination of probable paths for reaction; and IV, development of an experimental program. In Phase I, the major calculations were completed during the first year of the program, and further work awaits completion of other phases. In Phase II, most investigations of the molecular path of the steam-carbon reaction agree that the rate-limiting step in the reaction is the desorption of the product carbon monoxide, which involves splitting the carbon-carbon bond where the carbon monoxide is bound to the graphite crystallite. The major portion of the effort during the past year concerned the study of this bond. In Phase III, studies indicated that the rate-limiting step was the desorption of carbon monoxide from the graphitic crystallite. More literature treating this topic was studied, and the conclusion was formed that there may be complexities in this reaction that have not been treated. These complexities concern a possible surface-diffusion step in the reaction sequence. Nevertheless, it was also concluded that regarding the carbon monoxide desorption step as rate-limiting should remain the basis of the program for the present. In Phase IV some further study has been made concerning reactor design considerations for future experiments.

**PLANS FOR THE COMING YEAR** — The calculations and conclusions of Phase I, will be analyzed and documented. A major problem in Phase II is the fast relaxation of any excitation of the bond involved. This problem will be studied, with particular emphasis on the carbon-carbon bond. Calculations will be carried out in Phase III to determine if there is any purpose to exciting the gas-phase

species, that is, to see if an excited-gas species could stimulate another path to the product rather than the present one involving the rate-limiting desorption of carbon monoxide. Also, calculations will be made on how the surface-diffusion step may complicate the promotion of the reaction by the means under study. Once the other calculations and studies are completed, design of a Phase IV experimental program will be completed.

## ELECTROCHEMICAL GASIFICATION OF COAL

UNIVERSITY OF CONNECTICUT

DOE - \$40,000

9/77 - 8/79

Principal Investigator - R.W. Coughlin

**OBJECTIVES** – This project seeks to develop and demonstrate a novel approach to coal gasification using electrochemical technology.

**RECENT WORK AND ACCOMPLISHMENTS** – No effort or funds have yet been expended except to recruit suitable research workers.

**PLANS FOR THE COMING YEAR** – Research work will be initiated. Research personnel should be in place by December 1977.

## KINETICS AND MECHANISM OF DESULFURIZATION AND DENITROGENATION OF COAL-DERIVED LIQUIDS

UNIVERSITY OF DELAWARE

DOE - \$658,000; UD - \$238,000

6/20/75 - 6/19/78

Principal Investigators - J.R. Katzer, B.C. Gates

**OBJECTIVES** – This project shall determine reaction networks and reaction kinetics for catalytic hydrodesulfurization and hydrodenitrogenation of compounds found in coal-derived liquids; determine the relative reactivities of sulfur- and nitrogen-containing compounds and the important factors affecting their reactivity in coal-derived liquids; develop quantitative data on the chemical and physical properties of catalysts aged in coal-liquefaction processes; establish the mechanisms of deactivation; and develop reaction engineering information and models to predict the behavior of catalytic hydroprocessing of coal-derived liquids.

**RECENT WORK AND ACCOMPLISHMENTS** – The unique high-pressure flow reactors developed in this work and batch autoclave reactors have been used for the first quantitative determination of the reaction networks and kinetics of (1) catalytic hydrodesulfurization of dibenzothiophene and methyl-substituted dibenzothiophenes and (2) catalytic hydrodenitrogenation of quinoline, methyl-substituted quinolines, acridine, and carbazole. The catalysts were commercial hydroprocessing catalysts, i.e., sulfided  $\text{CoMoO}_3/\gamma\text{-Al}_2\text{O}_3$ ,  $\text{NiO-MoO}_3/\gamma\text{-Al}_2\text{O}_3$ , and  $\text{NiO-WO}_3/\gamma\text{-Al}_2\text{O}_3$ . At typical conditions of 300°C and 104 atm, dibenzothiophene reacted to give  $\text{H}_2\text{S}$  and biphenyl in high yield, but some hydrogenation accompanies hydrodesulfurization. The Ni- and W-containing catalysts gave relatively more hydrogenation than the Co-containing catalyst, but the important result is that all three catalysts had approximately the same activity and all were highly selective for sulfur removal. Methyl-substituted dibenzothiophenes react similarly to dibenzothiophene; each reaction is pseudo first-order in the sulfur-containing compound. Two methyl groups near the sulfur

atom double reactivity. These results are consistent with steric and inductive effects. In contrast, nitrogen-containing aromatics require hydrogenation of the aromatic ring before C-N bond rupture occurs. Saturation of the O-propyl aniline is required before nitrogen removal; propylcyclohexane is the primary hydrocarbon product.

The reaction network for carbazole is similar. Acridine and its homologs appear to be among the least reactive nitrogen-containing compounds in coal-derived liquids. An inventory of sulfur- and nitrogen-containing compounds is available for standards. Aged catalysts taken from the Synthoil and H-Coal processes and a proprietary fixed-bed process were analyzed with an electron microscope, with an electron microprobe, and by energy dispersive x-ray analysis. Deactivation occurs by coking, by mineral matter deposition in the interior and on the exterior of the catalyst, and by reductive deposition of iron and titanium in the interior of the catalyst. The ebullated-bed of the H-Coal process prevents deposition of mineral matter external to the catalyst by attrition.

**PLANS FOR THE COMING YEAR** – The research on hydrodesulfurization during the coming year will involve determination of the relative reactivities of four- and five-membered ring aromatic sulfur compounds, further quantification of the reaction kinetics of hydrodesulfurization, and evaluation of catalyst parameters affecting activity. The research on hydrodenitrogenation will involve determination of the relative reactivities, reaction networks, and partial kinetics for hydrodenitrogenation of four- and five-membered ring aromatic (acridine) nitrogen compounds, quantification of the kinetics and reaction network for carbazole, and determination of the interactions between nitrogen and aromatic, nitrogen and nitrogen, and nitrogen and sulfur compounds to provide insight into hydroprocessing reactivity of coal-derived liquids. The hydroprocessing reactor modeling studies will incorporate transient laydown of coke and mineral matter on catalysts and hydrodesulfurization and hydrodenitrogenation kinetics to predict reactor performance.

## COMBUSTION OF COAL-METHANOL SLURRIES IN THE HARTMANN RESONATOR

UNIVERSITY OF SOUTH FLORIDA  
DOE - \$20,000; University of South Florida - \$850  
9/15/77 - 9/14/78  
Principal Investigator - S.C. Kranc

**OBJECTIVES** – This program intends to develop an efficient combustor for coal slurries based on the Hartmann resonator. Methanol was chosen as the slurry medium because of the current interest in conveying coal in pipelines using liquids other than water. Methanol is volatile combustible and interacts with coal. Large loading ratios may be achieved, especially if fine particles are used. A second advantage results from the combustion of methanol-coal slurries. If the methanol is pure, then the total sulfur content of the mixture is lowered or else higher sulfur content coals may be used without exceeding SO<sub>2</sub> emission standards. The Hartmann resonator has been previously demonstrated as a combustor. The ultimate goal is to produce an efficient and high-intensity combustor for slurries. If the Hartmann resonator proves to be a practical device, then it could be directly applied to steam production or gas turbine combustion.

**RECENT WORK AND ACCOMPLISHMENTS** – An experimental facility for testing the resonator combustor is being constructed. The facility includes a high-pressure preheated air source, a flame tube, and associated monitoring instruments. Several adjustable models of the resonator combustor

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have been fabricated and evaluated for acoustic performance. Several operational modes have been observed. These models are now ready for atomization and combustion tests with pure methanol and then with slurries. A stirred reservoir and pumping system with positive displacement is being developed to handle slurry injection.

***PLANS FOR THE COMING YEAR*** – Development of the resonator combustor will continue. To assess the potential for this device, it will be necessary to study the operational characteristics parametrically in order to find a stable and efficient operating regime. Work will include studies of various slurry types. Both bituminous and lignite coals slurries will be tested. Eventually an exhaust gas sampling system will be added for analytic purposes. The results of these diagnostic measurements should yield combustion intensity and efficiency. If this work proves successful, further effort will be devoted to practical design and implementation.

### **TREATMENT OF PHENOLIC WASTEWATER WITH ANAEROBIC-ACTIVATED CARBON FILTERS**

GEORGIA INSTITUTE OF TECHNOLOGY  
DOE - \$37,800; Georgia Institute of Technology - \$11,966  
9/1/77 - 8/31/79  
Principal Investigator - M.T. Suidan

***OBJECTIVES*** – This research involves the development of an advanced wastewater treatment system capable of handling high strength phenolic wastewater and consisting of an anaerobic biological filter using activated carbon as a contact media. Phenols are present in appreciable concentrations in the liquid wastes generated by the coal gasification industry. The high and continuously fluctuating concentration of phenol renders the treatment of such wastewaters by conventional methods unreliable and unstable. The proposed process tends to stabilize the treatment system by the presence of activated carbon which acts as a buffer to store excessive concentrations of phenol and to release them back to the solution when the influent concentration decreases. The proposed process also produces methane rich gaseous products which could be utilized as an energy source within the plant.

***RECENT WORK AND ACCOMPLISHMENTS*** – Activities were centered around ordering equipment and materials and preparing shop drawings for construction of the experimental apparatus.

***PLANS FOR THE COMING YEAR*** – During the coming year, the reactor system will be constructed and experimental work initiated. The experimental apparatus consists of a series of individual fluidized-bed reactors that allow sampling at intermediate points to determine the rate of adsorption, desorption, and biodegradation of influent compounds. The reactors are fluidized by recycling the wastewater around each individual reactor. A batch reactor will also be operated to provide a constant source of acclimated seed for the column units. The first phase of the study is an investigation of the performance of the system when the influent waste contains a readily biodegradable substrate. The second stage will concentrate on the effect on the system's performance when varying the influent concentrations of phenol.



## SEPARATION OF PARTICLES FROM COAL-DERIVED LIQUIDS

ILLINOIS INSTITUTE OF TECHNOLOGY

DOE - \$56,316; Illinois Institute of Technology - \$14,448

8/1/77 - 7/31/79

Principal Investigators - D. Gidaspow, D.T. Wasan, W.M. Langdon, W.W. Waterman

**OBJECTIVES** — One of the major bottlenecks in development of coal liquefaction technology is the removal of fine solid particles after the liquefaction step. This program is developing an improved method of separation of coal-derived particles resulting from various liquefaction and solvent refined coal processes.

**RECENT WORK AND ACCOMPLISHMENTS** — Two new approaches to separation of particles from coal-derived liquids look promising. They include settling with use of a high-voltage electric field and separation of particles in a specially built cross-flow electrofilter. A cross-flow electrofilter was constructed and tested with a synthetic slurry. The slurry consisted of tetralin—a proton-donor solvent used in adding hydrogen to coal and alumina particles of size 0.3 to 2 microns. A clear filtrate was obtained at a strength of electric fields of the order of 1000 volts/cm. The parasitic losses from electrical conductivity were found to be very small. Thus, preliminary data indicate that the new device should be able to compete with pressure precoat filtration and hydroclones. Preliminary settling experiments for coal-derived slurries were carried out using samples received from four operating pilot plants—SRC, H-Coal, Synthoil, and Amoco.

**PLANS FOR THE COMING YEAR** — Cross-flow electrofiltration experiments will be conducted using samples from pilot plants diluted with xylene. A batch-settling apparatus to study settling at room as well as at elevated temperatures is being constructed. The particle concentration will be analyzed using a gamma-ray analyzer.

## CARBONYLATION STUDIES OF ILLINOIS COAL WITH CARBON DIOXIDE AND SODIUM METAL

ILLINOIS STATE UNIVERSITY

DOE - \$38,000; Illinois State University - \$16,506

8/1/77 - 7/30/79

Principal Investigator - R.C. Duty

**OBJECTIVES** — This study seeks to determine the solubility characteristics of Illinois bituminous No. 6 coal after it has been finely divided and treated with sodium metal and pressurized with carbon dioxide for prolonged reaction periods. The studies will be made at increasing increments of temperature from 50° to 250°C with carbon dioxide pressures to several hundred atmospheres. Carbonylation studies of coal have not been attempted previously, and this research should elucidate, in part, the basic structure of bituminous coal in regard to potential sites for generating aromatic radical anions, phenolic anions, and alkoxy anions. These aromatic radical anions and phenolic anions are potential precursors for generating carboxylic acids and phenolic carboxylic acids, respectively, from carbon dioxide. If large amounts of carbon dioxide are incorporated into the coal molecule as carboxylic acids, these ionic sites should increase the solubility characteristics of coal in aqueous and polar organic solvents. This study will ascertain, in addition to the solubility characteristics of carbonylated coal, the number of these aromatic anion sites generated as well as the number of phenolic and alkoxy anion sites produced.

**RECENT WORK AND ACCOMPLISHMENTS** – The solubility studies of bituminous coal at room temperature and atmospheric pressure have been measured and show a solubility of 4.63 percent in tetrahydrofuran and water. The coal was reacted with sodium metal in anhydrous tetrahydrofuran (THF) and washed several times with warm water after excess sodium was removed with wet THF. Coal, which was reacted with sodium metal in THF and subsequently treated with carbon dioxide at room temperature and atmospheric pressure, increased its solubility to 7.38 percent. Varying the mesh size of coal from 100 mesh (Tyler) to 400 mesh indicated no appreciable difference in solubility. Pressurized reaction with carbon dioxide and sodium metal at 50° and 150°C have been run in the autoclave as has the solubility of carbonylated coal, and the experimental data for these reactions is being analyzed. The solubility for the carbonylated coal at 50°C was 3.5 percent while the 150°C sample had increased the solubility to 9.5 percent. A partially completed material balance for the total sodium content by flame emission indicates the insoluble residue from the 50°C reaction temperature had increased its sodium content to 1.66 percent. This represents a ratio of 1 atom of sodium for every 83 carbon atoms, assuming the carbon content of the residue is 72 percent. The Illinois No. 6 coal sample has an average sodium content of 0.124 percent (neutron activation analysis by Illinois State Geological Survey) which corresponds to a sodium-to-carbon-atom ratio of 1:1110. A comparison with the 1:83 ratio reveals a thirteenfold increase in the sodium content of the coal which had been sodium treated and carbonylated with carbon dioxide at 50°C.

**PLANS FOR THE COMING YEAR** – The carbonylation studies with the bituminous coal will be continued at elevated temperatures and pressures, and solubility studies at 150°, 200°, and 250°C will be performed at several atmospheres of carbon dioxide pressures. Material balances at each temperature for total sodium consumed will be measured to ascertain the degree of sodium uptake by the water soluble coal samples and by the insoluble coal residues. All sodium analyses will be done with a flame emission spectrometer. Carboxylic acids generated will be analyzed by acid-base titrations, and phenolic and alcoholic sites will be determined by the acetic anhydride method. At each of the previously mentioned temperatures, the degree of solubility will be measured for the THF/water system along with the number of carboxylic acids, phenolic, and hydroxyl groups present in the soluble aqueous fractions and the insoluble solid residues. Sodium analyses, before and after the carbonylation studies, will ascertain the sodium uptake per 100 carbon atoms in the coal samples.

#### COMBUSTION STUDIES OF COAL-IN-OIL DROPLETS

NORTHWESTERN UNIVERSITY  
DOE - \$40,000; Northwestern University - \$2106  
8/1/77 - 7/31/79  
Principal Investigator - C.K. Law

**OBJECTIVES** – This project seeks to gain fundamental understanding of the vaporization and combustion mechanism of complex multicomponent hydrocarbon fuel droplets, emulsified with small amount of fine coal particles, in environments representative of various types of liquid-fueled combustors. A study is being carried out to ascertain whether the droplets vaporize in an essentially quiescent manner forming and exhausting large, unburned, coal particle agglomerates, or whether explosive vaporization induced by internal boiling is the actual vaporization mode resulting in non-formation of the agglomerates and in significant reduction of the overall level of fuel spray heterogeneity.

**RECENT WORK AND ACCOMPLISHMENTS** – For the theoretical phase, the equations governing the phenomena of interest have been established. For the experimental phase, the basic design for the free droplet generator has been conceptualized and fabrication has been initiated. A stream of free droplets is produced in the apparatus by a capillary tubing vibrating at the Rayleigh instability frequency and is injected into a chamber whose environment can be accurately controlled. The combustion process can be observed through high-speed cine-microphotography and stroboscopy.

**PLANS FOR THE COMING YEAR** – For the theoretical phase, the governing equations will be solved numerically. The composition and temperature of the droplet interior will be obtained, from which the location and the instant for the occurrence of the boiling state can be predicted as functions of the environment and of the initial droplet composition. For the experimental phase, the free droplet apparatus will be built, some initial experimentation conducted, and the explosive combustion will be studied.

### SOLIDIFIED NONEQUILIBRIUM ALLOYS AS FOSSIL FUEL CATALYSTS

SOUTHERN ILLINOIS UNIVERSITY-CARBONDALE

DOE - \$40,000

8/1/77 - 7/30/79

Principal Investigator - W. Brower

**OBJECTIVES** – This program intends to develop new catalytic systems for fossil fuel conversion. These new systems will be rapidly solidified, nonequilibrium alloys made by a splat cooling technique. This technique alters the lattice parameters of alloys and changes them into new catalyst systems. Such systems catalyze new reactions and open new pathways for fossil fuel conversion. On the other hand, systems that increase rates of present reactions would increase efficiencies of fossil fuel conversion, and systems that are made from low-cost raw materials would substantially lower the cost of fossil fuel energy.

**RECENT WORK AND ACCOMPLISHMENTS** – The splat cooling device that produces rapidly solidified, nonequilibrium catalysts has been designed, and its fabrication is nearing completion. Mating the splat cooling device with the induction heating power supply is progressing well. Initial heating experiments with the device show that temperatures in excess of 3000°C can be achieved. Hence, the splat cooling device has the ability of melting any of the proposed candidate catalyst alloy. Devices for controlling atmosphere and temperature during splat cooling have been ordered and some are being installed. High-purity alloying elements for the nickel-based catalyst systems have been purchased, and x-ray diffraction standards for comparison to splat cooled samples have been run.

**PLANS FOR THE COMING YEAR** – Construction and installation of the splat cooling apparatus will be completed, and the first series of candidate alloys will be evaluated. The alloys will be composed of a nickel-base with additions of silicon, tin, germanium, and gold. The silicon, germanium, and gold additions will expand the lattice parameter of nickel, whereas tin additions will shrink it. Such nonequilibrium solid solutions will exhibit lattice parameters not available in equilibrium structures. Meanwhile, x-ray diffraction and transmission electron microscopy data will characterize the structures of the splat cooled nonequilibrium solid solutions. Finally, evaluation of the catalytic activity of nonequilibrium structures will be conducted by determining their relative activity and selectivity in reactions of cis-cyclododecene with hydrogen and deuterium. Comparison

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of these activities and selectivities with those of corresponding equilibrium alloys will reveal any unusual catalytic properties toward activation of hydrogen. Future studies will test such identified, unusual, new catalyst systems in fossil fuel conversions.

### COAL ANION STRUCTURE AND CHEMISTRY

UNIVERSITY OF CHICAGO

DOE - \$207,000

3/77 - 3/80

Principal Investigator - L.M. Stock

**OBJECTIVES** – This research involves the development of new methods for coal alkylation, the use of these methods in the preparation of isotopically labeled soluble coal compounds, the characterization of the coal compounds by nuclear magnetic resonance spectroscopy, and the development of a satisfactory theory for the coal structure that accommodates the experimental observations. The realization of this objective will provide a more secure basis for the development of reagents and catalysts for coal conversion processes.

**RECENT WORK AND ACCOMPLISHMENTS** – The soluble alkylation products of a butylated Illinois No. 6 coal have been investigated by proton and carbon NMR spectroscopy. The results indicate that the four higher molecular weight fractions ( $MW > 5000$ ) have very similar highly paraffinic structures. The eight intermediate molecular weight fractions ( $450 < Mw < 5000$ ) contain molecules of very different structure. For example, the ratio of O-butylation: O-butylation ranges from 1.4 to 3.2 as the molecular weight decreases from 5000 to 450. The reductive butylation of aromatic fragments and the free radical butylation of aromatic rings are both important processes. O-butylation occurs exclusively on aryl oxygen atoms. Vinyl and formyl resonances are observed in selected fractions. The lightest fractions representing only 2.3 percent of the coal product are highly linear alkanes. The control experiments which are prerequisite to the use of reagents labeled with carbon-13 and deuterium have also been performed.

**PLANS FOR THE COMING YEAR** – The objectives for the next year include work on the use of new electron transfer agents in the presence of and in the absence of crown ether promoters, the synthesis of carbon and deuterium labeled reagents for the alkylation reaction, and the study of the products of the alkylation reagents using these specifically labeled reagents.

### MODELING OF A FLUIDIZED-BED COMBUSTOR WITH IMMERSSED TUBES

UNIVERSITY OF ILLINOIS AT CHICAGO CIRCLE

DOE - \$89,016; University of Illinois - \$28,670

5/8/75 - 9/9/78

Principal Investigator - S.C. Saxena

**OBJECTIVES** – This project intends to develop a mathematical model of a fluidized-bed combustor that will include coal combustion phenomena and will incorporate basic mass transport relationships, bubble mechanics, heat transfer, and configuration effects. A cold model test bed will be designed, constructed, and operated to generate data in support of the effort in developing the mathematical model. In particular, experiments will provide data concerning heat transfer effects of tubes and tube bundles in fluidized beds, bubble formation, dispersion etc. The successful completion of the analytical research will provide a reliable base for the design and scale-up of fluidized-bed combustors. The experimental work will enable the calculation of heat transfer by

immersed boiler tubes or their optimum design for a certain heat removal rate from the fluidized bed.

**RECENT WORK AND ACCOMPLISHMENTS** – A mathematical expression for the unsteady-state heat balance has been derived for a nonisothermal, noncatalytic, first order, gas-solid reaction. A theory has been derived for a quantitative treatment of solids projection from a dense fluidized-bed surface. World literature on heat transfer between a fluidized bed and immersed tubes has been critically examined including the fundamental mechanisms of wall-to-bed heat transfer. On the basis of the latter, criteria have been developed to evaluate the existing tube-to-bed heat transfer correlations. An extensive experimental program of research has been initiated to investigate the effect of fluidized-bed variables on heat transfer rate in a well instrumented and carefully designed square fluidized bed (0.305 by 0.305 m). Experimental results have been obtained on the total heat transfer coefficient between heated 12.7mm copper tubes and fluidized beds of different size glass beads, dolomite, silicone carbide, alumina and sand as a function of fluidizing velocity. In all cases, it has been found that the heat transfer coefficient first increases with fluidizing velocity up to a maximum value and then decreases with further increase in the fluidizing velocity. The initial rise has been attributed to the enhanced solids circulation and decreased particle residence time. The later decrease is due to the increase voidage or decreased particle packing density close to the heat transfer surface. The rate of heat removal is much larger for smaller particles and the heat transfer coefficient is independent of the value of the heat flux. Experiments have also been conducted to determine the influence of surface roughness on heat transfer coefficient. The comparison of results for the rough and technically smooth coefficient is strongly dependent on the ratio of pitch to particle diameter. By the proper choice of this ratio, the maximum heat transfer coefficient could be increased by as much as 40 percent over the value for a smooth tube. However, if this characteristic ratio is smaller than unity, the maximum heat transfer coefficient for rough tubes is smaller than the smooth tube.

**PLANS FOR THE COMING YEAR** – Experiments will be conducted with banks of tubes to investigate the effect of tube gap for different arrangements on heat transfer coefficient as a function of fluidizing velocity in a fluid bed of glass beads and sand of different particle diameters.

#### SEPARATION AND STRUCTURE ELUCIDATION OF COAL MOLECULE FRAGMENTS

INDIANA UNIVERSITY  
DOE - \$70,403; Indiana University - \$12,000  
2/1/76 - 1/31/78  
Principal Investigators - L.J. Todd, M.V. Novotny

**OBJECTIVES** – This project seeks to develop a glass capillary-gas chromatograph-mass spectrometry (GC-MS) method for the rapid separation and identification of molecules present in coal-derived liquids. The most efficient use of coal-derived liquids as an energy source of organic chemicals requires a detailed and rapid analysis of the types and quantities of different molecules present in any sample under consideration. The glass capillary-gas chromatograph-mass spectrometry method is one of the most promising approaches for this analysis.

**RECENT WORK AND ACCOMPLISHMENTS** – Solvent refined coal (without recycle oil) from the Pittsburg & Midway plant in Fort Lewis, Washington, was separated into acid, base polar neutral, polynuclear aromatic hydrocarbon (PAH), and aliphatic neutral fractions by the solvent partition

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scheme described previously. The PAH fraction was separated by glass capillary chromatography between 100° and 240°C and analyzed peak-by-peak with a Hewlett-Packard 5980A mass spectrometer. Characterized by molecular weight and formula were 146 components. Compound structures were assigned for almost all of these materials. The aliphatic fraction was separated on a 20m by 0.25mm glass column that was silylated prior to coating with OV-101, a nonpolar methylsilicone stationary phase. The GC-MS study between 100° to 270°C gave 33 fairly resolved peaks. These GC peaks contain two or more "nearly n-alkane" isomers each. These materials range in molecular weight between 170 and 394.

**PLANS FOR THE COMING YEAR** – The basic and acidic fractions obtained in the solvent partition scheme will be subjected to GC-MS analysis. The basic fraction will be chromatographed on a glass capillary column coated with UCON-50-HB-2000 polar polyglycol. The acid fraction initially will be separated into aliphatic acids, phenols, and aromatic carboxylic acids on DEAE-Sephadex A25 (a weak anion exchanger). The acids will be derivatized to obtain volatile materials before gas chromatographic separation. The separation and complete GC-MS analysis of the recycle process solvent (heavy distillate) from the SRC pilot plant in Washington will be performed. For the heavier PAH compounds (molecular weight 216 to 280) obtained from the SRC, the mass spectra were not useful in pinpointing specific structures for each component. Comparison of the retention times of known 4- and 5-ring derivatives with the retention times of the heavy SRC components should clear up many of the structural assignment difficulties.

## GASIFICATION IN PULVERIZED COAL FLAMES

PURDUE UNIVERSITY  
DOE - \$208,491; Purdue University - \$6254  
6/26/75 - 6/26/78  
Principal Investigator - N.M. Laurendeau

**OBJECTIVES** – This project investigates the feasibility of using available pulverized-coal burners to produce power (or synthesis) gas from coal. The configurations to be considered are the annular confined jet with secondary swirl and the vortex tube with tangential entry. Gasification of coal to synthesis and power gases is an economically and environmentally attractive means of expanded coal usage. Synthesis gas is a major feedstock for the production of methane, methanol, hydrogen, ammonia, and liquid hydrocarbons. Clean power gas may be used as an industrial fuel or, more importantly, in combined cycles, boilers, and MHD devices to generate electricity. This investigation is primarily concerned with the formation of a clean, low-Btu gas from coal. Entrained flow methods will be emphasized since they demonstrate significant advantages compared to fixed and fluidized-bed techniques.

**RECENT WORK AND ACCOMPLISHMENTS** – Design and construction of the cyclone gasifier is complete. The reaction chamber consists of a 6-in.-diameter by 10-in.-long cylinder. The pressure vessel was designed to operate at pressures up to 20 atmospheres. Pulverized coal and air enter the reaction chamber through four equally spaced tangential nozzles at velocities ranging from 100 to 300 ft/sec. A torch ignitor for chamber preheating and coal ignition fires downward from the chamber roof. Steam for gasification will be injected into the core of the reactor from the bottom. Separate coal/air and steam injection coupled with intense swirl will create an exothermic reaction zone in the outer annular region and an endothermic gasification zone in the core of the chamber. Product gas will exit through the top of the reactor and slag will be removed through the bottom.

Fabrication of the confined jet gasifier is underway. Test cell flow, heating, and control systems are installed and in working order. A series of methane combustion experiments were carried out to confirm the reliability of the torch ignitor and to check the flow metering and product gas analysis systems. Analytical modeling efforts are underway; the purpose is to interpret the combustion and gasification results and to provide insight into the nature of the processes involved.

**PLANS FOR THE COMING YEAR** – Coal combustion at atmospheric pressure in the cyclone reactor is scheduled for completion early in 1978. The confined jet gasifier will be installed and operated soon afterward. Systems for probing and traversing the reaction chambers will be designed and fabricated. Coal gasification at atmospheric pressure will follow. Work on analytical combustion and gasification models will continue.

### SULFUR FIXATION DURING COAL GASIFICATION

PURDUE RESEARCH FOUNDATION  
NSF/RANN/DOE - \$161,900; Purdue - \$8523  
3/75 - 3/78  
Principal Investigator - R. Schuhmann, Jr.

**OBJECTIVES** – This project intends to develop the basic scientific and engineering data for design of a coal gasification process in which the sulfur is fixed and separated in chemical combination with iron. A further objective is laboratory-scale testing of the feasibility of the combined gasification-sulfur fixation process in a fluidized-bed reactor. The sulfur content of the product low- or medium-Btu gas must be low enough that the gas can be used for power and steam generation within acceptable limits of sulfur emission.

**RECENT WORK AND ACCOMPLISHMENTS** – Thermodynamic calculations and phase equilibrium studies have shown that iron oxides, metallic iron, and iron silicates should be effective sulfur fixation agents over wide ranges of gasification temperatures and gas compositions, both below and above the melting point of iron sulfide. The thermodynamic evaluation also showed that iron should be especially effective for sulfur fixation from gases with low  $H_2/CO$  ratios. Specific reaction rates and rate constants for sulfur fixation with iron have been measured experimentally as a function of temperature and iron oxide particle size. For particle sizes below 10 mesh and temperatures above  $825^{\circ}C$ , the measured reaction rates are sufficient to justify the combination of gasification and sulfur fixation in the same fluidized-bed reactor. Laboratory scale tests of a top-blown closed-bottom jet-fluidized reactor have shown that a highly turbulent fluidized bed of coke or char can be maintained above molten slag and metallic phases. The results of early tests of combined gasification and sulfur fixation in a 3-in.-diameter jet-fluidized reactor are promising.

**PLANS FOR THE COMING YEAR** – An improved fluidized-bed reactor capable of sustained steady-state operation will be constructed for combined char gasification and sulfur fixation with iron. This reactor will be used to obtain further kinetic and reaction rate data on sulfur fixation with iron, as well as other data relevant to the feasibility of the combined process.



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## MICROSTRUCTURAL EFFECTS IN ABRASIVE WEAR

UNIVERSITY OF NOTRE DAME

DOE - \$109,267; UND - \$5223

3/14/77 - 3/13/79

Principal Investigator - N.F. Fiore

**OBJECTIVES** – This program intends to establish quantitative relations between microstructure and abrasive wear under gouging and low-stress conditions for a number of alloys used in coal mining, handling, and gasification. The relations will allow an empirical ranking of the wear resistance of the alloys and will also provide the designer and materials engineer with rules, based on the principles of physical metallurgy, for the specification of materials for wear resistance. The work is being conducted on white irons and Co-base superalloys. The program is based on a three-faceted experimental approach involving wear testing, mechanical testing, and metallography.

**RECENT WORK AND ACCOMPLISHMENTS** – Standards were selected for the low-stress rubber-wheel abrasive tested (RWAT) and the gouging,  $\text{Al}_2\text{O}_3$  wheel abrasive tester (GAW). Both annealed AISI 1020 steel and quenched and tempered ( $\text{R}_c 52$ ) vacuum-arm remelted AISI have been tested. Minimum variability in abrasion factor (weight loss of test sample vs weight loss of standard) is provided by 1020 steel, so this material has been selected as the standard. The RWAT tests on the white irons indicate that microstructure has a marked effect on wear. For the case of the low-Cr white irons, weight loss for the 2.7 percent pearlitic iron is 30 percent greater than that of the 3.5 percent pearlitic iron, because the higher-C iron has a larger volume fraction of the wear-resistant carbide phase  $\text{Fe}_3\text{C}$ . In addition, for either iron, the weight loss normal to dendrite axes is about 20 percent greater than that in the plane of the axes. Thus, both volume fraction and shape of phases influence abrasion resistance. The 27 Cr iron with its extremely hard  $\text{Cr}_7\text{C}_3$  carbides and martensitic matrix displays the best RWAT abrasion resistance. Retained austenite ( $\gamma$ ) is intrinsically soft, so that as its volume fraction increases from 5 to 85 percent, the material hardness decreases from 65 to 50 Rockwell C. It is evident that wear is a strong function of retained  $\gamma$ , and that intermediate amounts of  $\gamma$  produce poorest wear resistance. It is also evident that the RWAT test results correlate with the pin test results from the AMAX laboratories, a finding which indicates that the RWAT system is providing a valid test of microstructural effects.

**PLANS FOR THE COMING YEAR** – The RWAT tests on the cast irons have been completed, and the microstructural effects identified. The microtopography of the wear scars will be measured and features on the worn surfaces related to microstructure. The objective of this portion of the program is to delineate the metal deformation-fracture processes controlling wear.

The GAW tests on the cast irons will be completed and results compared with the RWAT results. The Co-base alloy powder metallurgy samples will be subjected to RWAT and GAW testing. The microstructural effects observed in the cast irons have been so marked that a more in-depth study into their nature will be undertaken. The study will be performed on a nominal 20-Cr iron which is ideal for basic microstructural studies and is also of increasing practical importance in mining, handling, and direct utilization of coal. Market forecasts are for worldwide sales of 300 million t/yr of the alloy into the foreseeable future. The composition of various samples will be adjusted to provide continuously increasing carbide volume fraction at a constant matrix composition and microstructure. At one given carbide volume fraction, samples of the iron will be heat-treated to provide a series of matrix microstructures. In this manner, both the effect of carbide phase and matrix on wear will be determined.



## PERSPECTIVES IN ENERGY: 1977

KANSAS STATE UNIVERSITY  
DOE - \$12,700  
Principal investigator - J.K. Shultis

**OBJECTIVES** – This project will provide 30 junior high school, high school, and junior college teachers in the mid-plains regions with facts on and an assessment of the many facets of the national energy situation during a 5-day intensive workshop.

**RECENT WORK AND ACCOMPLISHMENTS** – During the past 6 summers, eight workshops have been conducted by KSU's Department of Nuclear Engineering. It has been found that a small workshop staff of highly qualified technicians is preferable to a large staff, each presenting one or two lectures. With a small staff covering most lecture areas, better rapport is established with the participants, group discussions are more productive, and less overlap occurs between lectures.

**PLANS FOR THE COMING YEAR** – A proposed 10-day summer workshop in general energy education has been designed to give the participants an overview of the energy dilemma with emphasis placed on topical subjects. In particular, those questions of special concern in the mid-plains region will be emphasized (e.g., agricultural aspects, wind systems, biomass conversion and coal gasification, state energy plans).

## CATALYTIC ACTIVITY OF COAL MINERAL MATTER

UNIVERSITY OF KENTUCKY RESEARCH FOUNDATION  
DOE - \$94,003; IMMR - \$15,113  
1/1/76 - 3/31/78  
Principal Investigator - C.E. Hamrin, Jr.

**OBJECTIVES** – This project intends to determine the ability of naturally occurring mineral matter found in coal to desulfurize and denitrogenate catalytically model sulfur- and nitrogen-coal compounds. A pulse microreactor connected to a gas chromatograph is the reaction system to be used. Mineral matter is to be obtained by low-temperature ashing of coal in an oxygen plasma. All liquefaction processes under development, catalytic or non-catalytic, add hydrogen to coal containing mineral matter. Depending on the process, hydrogen is added to increase the H/C ratio of the product and/or to remove heteroatoms such as S, N, and O. Successful completion of this study will rank the mineral matter of 8 to 10 U.S. coals in ability to desulfurize and denitrogenate model S and N compounds. In addition, the importance of the individual components and synergistic interaction among them will be determined. Modification of activity by mild chemical treatment and during either the SRC or SRL process will also be measured.

**RECENT WORK AND ACCOMPLISHMENTS** – Nine coals were selected and low-temperature ash

A statistical analysis of the conversions versus the XRF values showed potassium to give the best correlation with an R-value of 0.62. An even higher R-value of 0.87 was found for the surface area-conversion correlation. Of the clays tested, montmorillonite No. 22 was the most active, and its activity exceeded that of the Homestead LTA by 67 percent.

**PLANS FOR THE COMING YEAR** – Denitrogenation activity will be measured for the LTA's and SRC/SRL solids from various points in the process. In addition, a series of runs with  $\text{FeS}_{1+x}$  compounds will be made to see if there is an optimum value of  $x$  between 0 and 1 for catalyzing thiophene HDS.

## ANALYSIS OF DESIGNS FOR COAL CONVERSION PRESSURE VESSELS

UNIVERSITY OF KENTUCKY

DOE - \$246,699; UK - \$17,302

3/1/77 - 2/28/79

Principal Investigators - D.C. Leigh, T.R. Tauchert

**OBJECTIVES** – Although it appears that demonstration and first-generation coal gasification plants will be designed using shop-fabricated single-wall steel pressure vessels of about 12-ft-diameter, it is anticipated that as commercial feasibility is established, there will be a demand for much larger diameter (25 to 35 ft) field-fabricated vessels. When pressures of up to 1500 psi for coal gasification processes are combined with the larger diameters, the required wall thickness would be beyond single-wall steel technology, especially for field fabrication. Further, single-wall technology may be exceeded for coal liquefaction processes where pressures of 3000 or 4000 psi may be combined with diameters leading to relatively thick walls. The objective of this project is to conduct a comprehensive study of alternative structural designs for large diameter pressure vessels for various coal gasification and liquefaction processes. Alternative designs will be analyzed and evaluated critically on the basis of structural integrity, reliability, safety, and manufacturing parameters and constraints; and finally, optimized and compared on the basis of minimum overall cost. Work shall consist of the following tasks: Task I - Definition of Pressure-Vessel Needs; Task II - Preliminary Design and Analysis, an evaluation and comparison, including limiting constraints, of many types of structural-materials designs and a selection of a few of the most feasible; Task III - Advanced Design and Analysis, detailed designs will be done for selected combinations of structural designs and processes; Task IV - Determination of Manufacturing and Other Costs; Task V - Design Optimization, optimization shall be on the basis of total minimum cost, subject to constraints, and shall consist of two facets: the selection of the optimum structural-material combinations corresponding to the given sets of pressure vessel needs and the optimization of the selected structural-material combination; and Task VI - Recommendations.

**RECENT WORK AND ACCOMPLISHMENTS** – An extensive computer literature search has been done for information published since 1970 on pressure vessels including various types of structures and materials, designs and analyses, limitations, and cost. Also, a search was done on coal gasification and liquefaction for literature published during the past year. Over 2000 titles with abstracts were obtained; they have been reviewed and sorted and approximately 400 articles have been acquired. Task I, a structural classification of pressure vessels, has been defined along with a brief description of each. Information about the following types of pressure vessel structures was obtained from visits to fabricators, organizations, and technical society meetings: single-wall shrunk fit, concentric layered arcs, spiral layered arcs, prestressed concrete, prestressed cast iron, and some

information on spiral sheet and helically wound interlocking strips. An appreciation of the magnitude of materials problems in coal conversion technology was gained as well as information on designs for large-diameter coal gasification pressure vessels using single-wall and prestressed concrete constructions. Task II is well underway. Each type of pressure vessel structure will be discussed. Computer programs for the determination of stresses resulting from elastic and thermal effects in cylindrical layered walls have been adopted and some sample calculations completed. The formulation of exact and approximate methods of stress analysis for cylindrical vessels under stationary, nonaxisymmetric temperature distributions is underway. Consideration is being given in this study to temperature-dependent behavior, anisotropy, and the effects of creep. The results of the investigation will be useful in the Task III.

**PLANS FOR THE COMING YEAR** – Task II will be completed in the first quarter of the fiscal year; Task III should be completed for most of the designs; Task IV will be acquired during the year; and most of Task V will be completed.

## **SURFACE STRUCTURE AND MECHANISMS OF GASIFICATION CATALYST DEACTIVATION**

UNIVERSITY OF KENTUCKY  
DOE - \$391,286; UK - \$175,286; Catalysts and Chemicals, Inc. - \$77,131  
2/1/76 - 1/31/79  
Principal Investigator - P.J. Reucroft

**OBJECTIVES** – This research program seeks to obtain a detailed understanding of the factors governing the high specificity of nickel-based catalysts in methanation reactions; characterize and obtain a better understanding of the microscopic phenomena that lead to deterioration of catalyst performance through chemical poisoning; evaluate the validity of accepted models of catalyst thermal deactivation; and use the information obtained from the basic research program to design new catalyst systems with improved activity, selectivity, resistance to poisons, and thermal stability compared to available commercial catalysts.

**RECENT WORK AND ACCOMPLISHMENTS** – After extensively investigating coprecipitated nickel methanation catalysts (metal loading, 10 to 50 percent by weight) by the ESCA technique, studies are now being directed toward catalysts that have been employed in methanation reactors. Active catalysts with good methanation activity and sulfur deactivated catalysts have been examined. X-ray diffraction techniques for determining catalyst particle-size distributions have been improved and refined in a series of studies on nickel oxide dispersed in alumina and silica. Studies are now being focused on determining the effects of time and temperature on the particle-size distribution in reduced coprecipitated catalysts to gain insight into the thermal sintering mechanism. Studies on chemical bonding in several alumina and silica supported coprecipitated nickel methanation catalysts have emphasized the initial use of IR spectroscopy. Fresh catalysts, poisoned (deactivated) catalysts, and catalysts that have been employed in methanation reactors have been examined. These materials are being investigated by laser Raman and IR spectroscopy. IR spectroscopy has been used to investigate atom distributions in both fresh and used catalyst

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## HOT GAS DESULFURIZATION

UNIVERSITY OF KENTUCKY

DOE - \$155,158; UK - \$40,926

4/1/76 - 3/31/77

Principal Investigator - J.T. Schrodt

**OBJECTIVES** — This project seeks to develop and critically evaluate a high-temperature coal-derived fuel gas desulfurization process that utilizes gasifier coal ashes as sulfur sorbents. Efforts are focused on gathering bench-scale sorption data and formulating models suitable for the preliminary design and analysis of both fixed- and fluid-bed systems. A comparison of high- and low-temperature fuel gas usage in combined-cycle gas turbine and steam-boiler power plants indicates higher efficiencies can be achieved using hot fuels. Gasifier technology has progressed to the stage of full development and demonstration; however, high-temperature fuel gas desulfurization remains an area requiring further research and development. The hot ash process effectively removes hydrogen sulfide and carbonyl sulfide from coal-derived fuel gases at gasifier exit temperatures and pressures by utilizing hot, waste ashes from coal gasifiers.

**RECENT WORK AND ACCOMPLISHMENTS** — So far, 134 desulfurization-regeneration tests have been completed in small fixed-beds of four selected gasifier coal ashes. These ashes contain from 5 to 23 percent iron oxide. Results show sulfide removal efficiencies in excess of 98 percent and ash capacities of 400 grains of sulfide/lb of ash. Fully sorbed ashes regenerated up to 20 times using air at 1200°F show improved performances and increased capacities during the first 10 cyclic uses, which is associated with the transport of iron to the particle surfaces where it becomes readily available for reaction. Sorbents retain their capacities beyond 20 cyclic uses. At high temperatures, even dilute concentrations of hydrogen sulfide severely corrode most steels. Armco 18-SR shows good resistance to this environment. A fixed-bed process model has been computer programmed. It uses three dimensionless rate parameters to predict the concentration of hydrogen sulfide and available unreacted iron oxide as a function of time and position within a fixed-bed. The three parameters represent bulk gas-diffusion, gas pore diffusion, and uniform gas-solid chemical reaction. Sets of constant-pattern breakthrough data have been used to correlate these parameters with the independent physical and chemical properties of the system. Important properties are temperature, pressure, particle diameter, space velocity, and active iron concentration. The model, which is applicable to both the desulfurization and regeneration stages, and typical low-Btu fuel gas streams sufficient to supply a 1000 Mw power station operating at 40 percent efficiency are being used for design of a hot-ash, fixed-bed desulfurization process. The flowsheet and overall material and energy balances on this process are complete. Process design of the primary units is underway.

**PLANS FOR THE COMING YEAR** — The experimental system has been adapted and is carrying out fluid-bed desulfurization tests. A second model based upon the steady-state operation of separate desulfurization and regeneration fluid-bed units has been programmed. Work will focus on gathering additional data and reducing it to a form suitable for preliminary design of a full-scale fluid-bed system.

## THERMO-MECHANICAL MODEL FOR REFRACTORY CONCRETE LINER-ANCHOR INTERACTIONS

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

DOE - \$122,983

7/1/77 - 6/30/79

Principal Investigators - O. Buyukozturk, J.J. Connor

**OBJECTIVES** – This project is striving to develop an understanding of the effect of stainless-steel anchors on single- and dual-refractory liners of cylindrical coal gasification vessels during heatup and cooldown of these process units. A mathematical model will be devised to predict the stress strain distributions that may lead to cracking, creep, and other mechanical degradation processes. Analytical sensitivity studies on such a model would lead to design recommendations regarding heating schedules, material property combinations, and anchor spacings to avoid local degradation caused by anchor-liner interactions.

**RECENT WORK AND ACCOMPLISHMENTS** – Work is in progress to study the material behavior of monolithic refractories and to incorporate this behavior into a three-dimensional nonlinear, finite element program. Critical features of the program have been tested on simple verification problems. Several areas dealing with the mechanical stress analysis have been improved. Analysis features for temperature effects are being tested. Necessary material properties for refractory concrete have been obtained. Several material models are being considered for incorporating the capability of time and temperature dependent deformations.

**PLANS FOR THE COMING YEAR** – A heat transfer computer program will be adopted for determining temperature distributions through the thickness of cylindrical monolithic refractories. Necessary improvements will be made to both stress and heat transfer analysis capabilities. Parametric studies of the selected, finite element models will be performed for the effects of heating rates, geometrical parameters, and material property combinations. Based on these studies, design recommendations will be made to avoid or reduce the cracking and local degradation of monolithic refractory linings.

## PHYSICO-CHEMICAL PROPERTIES OF COAL SLAGS

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

DOE - \$183,900

9/1/77 - 8/31/80

Principal Investigators - J.F. Elliott, G.J. Yurek

**OBJECTIVES** – This program will assemble pertinent data on the thermodynamic behavior of the inorganic constituents in coal and the related constituents in coal ash. These data and other information on the properties of the constituent species of slags will develop correlations for predicting the thermodynamic properties of the components of polymeric- and ionic-type slags. Particular attention will be directed to the properties of potassium in these slags. The study will also be directed to developing correlations for predicting the equilibrium vapor pressures of species in coal slags and the rates of evaporation of these species.

**RECENT WORK AND ACCOMPLISHMENTS** – The initial month of the program was devoted to collecting information from the literature related to the thermodynamic properties of important compounds pertinent to the inorganic species that may be present in coal slags, coal ash, fly ash,

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and the gas phase in coal combustion systems. Thus far, data on the standard Gibbs' free energies of formation of the solid, liquid, and gaseous compounds in the Si-O system, the Na-S-O-H system, the K-S-O system and the Fe-O-H system have been collected and evaluated.

**PLANS FOR THE COMING YEAR** — A complete listing with abstracts and summary diagrams of sources in the literature related to the thermodynamic properties of the important components of coal slags will be compiled. This work will be closely related to the ranges of the inorganic constituents of coals that are to become available through pertinent information in the Penn State DOE Coal Sample and Data Banks. It will focus principally on systems containing iron oxides, silica, alumina, lime, potassium oxides, and related sulfates. Work will be continued to bring together thermodynamic properties of all of the solid, liquid, and gaseous compounds of interest in a common and consistent form. Means will be established for predicting the thermodynamic properties of major and minor constituents, with emphasis on potassium compounds of coal slags and coal ash based on the thermodynamic properties and phase diagrams that will be reviewed, collated, and compiled initially.

## **BASIC STUDIES OF COAL PYROLYSIS AND HYDROGASIFICATION**

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

DOE - \$250,707

1/1/77 - 6/30/78

Principal Investigator - J.B. Howard

**OBJECTIVES** — This research will provide an improved fundamental understanding of the conversion of coal to clean fuels by processes based on rapid pyrolysis and hydrolypyrolysis. Specific objectives are to measure compositions and rates of formation of products from the pyrolysis and hydrolypyrolysis of a partially dried Montana lignite and a Pittsburgh seam bituminous coal under systematically varied conditions pertinent to practical interest, including heating rates ( $65^{\circ}$  to  $12,000^{\circ}\text{C/s}$ ), pressures (0 to 100 atm of hydrogen, helium and mixtures thereof), final temperatures ( $400^{\circ}$  to  $1100^{\circ}\text{C}$ ), and particle sizes (5 to  $1000\ \mu\text{m}$ ), using small-scale batch and entrained-flow reactors. Other objectives are to develop correlations and predictive models from the experimental data and to determine the distributions of original sulfur and nitrogen in product chars.

**RECENT WORK AND ACCOMPLISHMENTS** — Significant accomplishments include completion of construction of the laminar flow reactor and extensive systematic studies of product compositions from the pyrolysis and hydrolypyrolysis of both coals using the batch reactor. Interesting results include identification of tradeoffs in the effects of temperature, time, and hydrogen pressure on the yields of methane and other products or classes of products in rapid hydrolypyrolysis. In addition, hydrogen at 69 atm in short residence time exerts a significant effect on methane yields from both lignite and bituminous coal at relatively low temperatures, such as  $600^{\circ}\text{C}$ , but high temperatures are required for an effect on total volatiles yield. In studies of short-residence-time pyrolysis, the principal volatile products from bituminous coal were gaseous hydrocarbons, tars, and liquids with only small yields of carbon oxides while the lignite gave high yields of water (pyrolytically formed) and carbon oxides.

**PLANS FOR THE COMING YEAR** — Research will include systematic studies of pyrolysis and hydrolypyrolysis using the entrained-flow reactor with emphasis on the effect of coal particle sizes in the range 74 to  $5\ \mu\text{m}$  on rates and extents of conversion to specific products and on the fate of

coal-bound sulfur and nitrogen under rapid heating conditions. The batch reactor will be employed to further expand the data base on rapid pyrolysis and hydropyrolysis and to examine in more detail the most promising reaction conditions previously identified.

### COAL PYROLYSIS BY HOT SOLIDS FROM A FLUIDIZED-BED COMBUSTOR

MASSACHUSETTS INSTITUTE OF TECHNOLOGY  
DOE - \$119,995; MIT Sloan Research Fund - \$10,000  
6/1/77 - 5/31/79  
Principal Investigator - J.P. Longwell

**OBJECTIVES** – This research intends to determine the technical feasibility of generating relatively clean, economically storable liquid and/or gaseous fuels by the fluidized-bed pyrolysis of coal in the presence of lime and/or other inorganic solids capable of in-situ  $H_2S$  and  $CO_2$  removal. Specific objectives are to measure yields of gaseous, liquid and solid products, gas compositions; and sulfur and nitrogen contents of chars and of successive distillation fractions of liquids from at least two coals of commercial importance. Pyrolysis is carried out in a fluidized bed (2-in. ID) in the presence of lime under conditions of practical interest including bed temperature ( $800^{\circ}$  to  $1600^{\circ}F$ ), total external pressure (1 to 20 atm), coal feed rate (0.5-5 lb/hr), lime-to-coal ratio (0-50), coal and lime particle-size distributions (250 to 500  $\mu m$ ), and fluidizing velocity (0.3 to 2 ft/sec). Related objectives are to develop global correlations and predictive models using these data. A preliminary engineering and economic assessment of the process will be performed by an industrial subcontractor.

**RECENT WORK AND ACCOMPLISHMENTS** – The design, construction, and testing of the fluidized-bed reactor and related product collection equipment have been completed. Material balances in the range 94 to 98 percent have been obtained with Montana lignite, a noncaking coal. Chromatographic methods for analysis of gaseous products as well as procedures for fractional distillation of small quantities of product liquids are under development. Other studies are focused on separation of char from mixtures containing sand and other solids used in the fluidized-bed. All preliminary test runs with Illinois No. 6 bituminous coals have resulted in severe caking and subsequent plugups. Suitable operating conditions for handling such caking coals are being studied. The development of predictive models for the performance of the bed and eventual scaleup of the process is also in progress.

**PLANS FOR THE COMING YEAR** – Systematic data collection will begin using Montana lignite as soon as acceptable protocols for gas analysis are completed. Regular data collection will ensue, using Illinois No. 6 bituminous coal, as soon as the determination of suitable operating conditions for handling caking coals is completed. The predictive models or performance equations under development will be tested and modified with the experimental data.

### MEDIUM- AND HIGH-TEMPERATURE GAS CLEANUP OF PARTICULATES

MASSACHUSETTS INSTITUTE OF TECHNOLOGY ENERGY LABORATORY  
DOE - \$150,000  
9/27/77 - 9/26/79  
Principal Investigators - J.F. Louis, J.R. Melcher

search program is an experimental investigation of the applicability of the  
B) to the collection of particulate products of coal combustion. Bench-scale

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experiments are to span the range of 400<sup>o</sup> to 1800<sup>o</sup>F covering conventional utility, fluidized-bed combustion systems, gasifier, and combined-cycle system operating regimes. Particulate injection will allow injection of combustion products of a variety of coals or even combustible material if desired. From this program, suitable models for predicting the behavior of high-temperature EFB's and workable designs will evolve.

**RECENT WORK AND ACCOMPLISHMENTS** – The design of the test rig has been completed and construction has begun. The test rig is being fabricated in stainless-steel sections of 9-in.-by-9-in. cross-section with external thermal insulation. Natural gas burners will supply hot combustion gases which, along with injected particulate, will pass through the EFB.

**PLANS FOR THE COMING YEAR** – The test rig will become operational, charging techniques for the particulate will be developed, and materials for the EFB construction will be selected and tested. By the end of the year, many of the operational characteristics of the high-temperature EFB should be outlined.

### ENERGY INSTITUTE FOR TEACHERS

SOUTHEASTERN MASSACHUSETTS UNIVERSITY

DOE - \$15,400

1/1/77 - 12/31/77

Principal Investigator - C.A. Smith

**OBJECTIVES** – The Institute was designed to help high school teachers of humanities and social studies gain a broad understanding of the physics and economics of energy problems and acquaint them with some of the humanistic resources that can be used to introduce energy problems into social studies and humanities classrooms.

**RECENT WORK AND ACCOMPLISHMENTS** – The Institute, conducted during the early summer of 1977, was attended by 25 teachers. No attempt was made to conduct research into energy problems or into the teaching of energy problems. The objective of helping the teachers who attended to understand energy problems better was achieved and through the effect on their teaching, two further objectives have been achieved. First, students will get more information and better understanding of the energy choices facing us. Secondly, and perhaps even more importantly, the students who encounter informed and relevant consideration of energy problems in their social studies and humanities classrooms will come to see that energy is not a problem that can be left to scientific experts. Because many of the most important energy decisions are political in nature, it is important that a reservoir of informed thinking be built up among the non-scientist majority in the electorate.

**PLANS FOR THE COMING YEAR** – It is not known whether a contract for a second Institute in the summer of 1978 will be awarded. Should it be, an attempt will be made to bring in some of the basic biology of the problems associated with the use of energy.



## CATALYTIC HYDROLIQUEFACTION/HYDROGASIFICATION OF LIGNITE

WORCESTER POLYTECHNIC INSTITUTE

DOE - \$142,000; WPI- \$8000

9/77 - 9/80

Principal Investigators - W.L. Kranich, A.H. Weiss

**OBJECTIVES** – The research project is aimed at investigation of the basic chemical kinetics and mechanisms in catalytic hydrogenation of lignite. Specifically, the research is designed to study the overall conversion rate and the elemental transformation from the raw materials into products under surface reaction conditions and to postulate the reaction mechanisms of hydroliquefaction and hydrogasification of lignite based upon the results obtained. A better understanding of continuous lignite conversion processes should thus be obtained which may lead to an improved ability to predict the behavior of pilot or demonstration plants under altered operating conditions and to possible directions for process improvements.

**RECENT WORK AND ACCOMPLISHMENTS** – Effort has been devoted to the recruiting of research personnel. Plans are being made for equipment modification and procurement.

**PLANS FOR THE COMING YEAR** – A high-pressure continuous stirred-tank hydrogenation unit based on equipment now in the WPI laboratories will be designed and built for the study of hydrogenation of lignite slurried in a carrier oil. Operating procedures will be established and tested and screening of catalysts will be undertaken. Methods for measuring and characterizing liquid and gaseous products will be developed and tested.

## PHYSICAL AND CHEMICAL BEHAVIOR OF LIQUEFIED COAL IN SOLIDS SEPARATION

UNIVERSITY OF MICHIGAN

DOE - \$378,768; UM - \$45,693

9/30/76 - 7/31/79

Principal Investigator - D.E. Briggs

**OBJECTIVES** – This project will study the physical and chemical behavior of liquefied coal. The interactions between various fractions of liquefied coal, solvents, mineral matter, and catalysts will be measured experimentally to provide solubility, colloid behavior, adsorption, viscosity, and surface tension data for the design and operation of coal liquefaction processes. Model compounds will be synthesized and examined in combinations to simulate properties of coal liquids. The anticipated data should contribute to the design and operation of future coal liquefaction pilot and demonstration plants, especially in setting the process conditions in the preheater-reactor and solids separation. Indications for improved handling and disposition of undesirable N- and S-containing components are anticipated.

**RECENT WORK AND ACCOMPLISHMENTS** – Four bis-(hydroxyphenyl) alkanes and four  $\alpha,\omega$ -dipyridyl or diquinolyl-alkanes were synthesized, purified, and examined by proton NMR. The compounds are believed to possess characteristics of asphaltenes. Solvent extracted asphaltenes from an H-Coal liquefaction of an Illinois No. 6 Coal were fractionated in a 10.16-cm by 122-cm long GPC column. Fractions were characterized by molecular weight, elemental, and proton NMR analyses. Oxygen and metals levels were determined by neutron activation. Exploratory experiments to separate asphaltenes into acidic and basic fractions were conducted. Filtration data

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were taken at 232°C at different concentrations of oils, resins, asphaltenes, and preasphaltenes with 13.8 wt percent THF insoluble solids. At comparable asphaltene and preasphaltene concentrations, the specific cake resistance is less with oils and resins than with tetralin. The colloid micelles that form in tetralin above 16 to 18 percent asphaltenes at 117°C are substantially peptized by resins. Electrophoresis experiments with asphaltene and preasphaltene fractions gave deposition on both electrodes, being largest on the positive electrode with pyridine, THF, and m-cresol and on the negative electrode with benzene and tetralin as solvents. Adsorption characteristics of GPC fractionated asphaltenes were examined. Streaming potential measurements were made for the flow of asphaltene solutions through fixed beds of solids to assess the effects of concentration, temperature, solvent, and solid adsorbent. There is consistency in the electrophoresis, adsorption, and streaming potential data.

**PLANS FOR THE COMING YEAR** — Work will continue on the synthesis and interaction of bis-(quinolyl) alkanes and polymethylene-bisphenols. The colloid micelle size in asphaltene and preasphaltene solutions will be measured by small-angle x-ray techniques. Filtration experiments will be conducted to measure the compressibility of THF insoluble solids at and above 232°C. Experiments will be made to determine the effect of selected additives on the filtration rate of liquefied coal. Procedures will be evaluated and developed for separating asphaltene and preasphaltenes into acidic and basic fractions and for determining the phenolic oxygen and basic nitrogen content of asphaltene and preasphaltene fractions. A method will be developed to effect asphaltene adsorption experiments in the 150 to 370°C range. The viscosity of a number of characterized asphaltene and preasphaltene samples will be measured separately and after mixing and analyzed statistically.

## RHEOLOGY STUDIES OF COAL-OIL SLURRIES

WAYNE STATE UNIVERSITY

DOE - \$40,000

9/1/77 - 8/31/79

Principal Investigators - S. Carmi, K.A. Kline

**OBJECTIVES** — This project seeks to develop a practical method for determining the rheological properties of coal-oil mixtures under various concentration and temperature conditions. A second objective is to determine pre-combustion atomization characteristics of the coal-oil slurry for different pressures and nozzle configurations. The research project addresses itself to a novel and direct coal utilization process in which pulverized coal is mixed with oil and burned in packaged oil-fired boilers. A significant problem in this process is maintaining a stable slurry, and very little is known concerning the mechanics of suspension stability, atomization, and spray formation of this non-Newtonian mixture. Direct characterization of the coal-oil slurry will enhance early detection of slurry instability and trigger corrective action for improved spray formation and combustion efficiency.

**RECENT WORK AND ACCOMPLISHMENTS** — As part of the fundamental rheological study, effort is concentrated in developing a non-Newtonian fluid model that will include an appropriate constitutive equation for the mixture. Preparations are also made to carry out experimental viscometric studies. A rotating rod viscometer is being built to help assess rheological properties of the mixture by studying the normal stress effect evident in non-Newtonian fluids. More viscosity measurements will be carried out at both the Wayne State University and the GM Technical Center

Laboratories using capillary rotating concentric cylinders and cone-plate viscometers and related sensing and recording instruments. Tests will be performed at a variety of concentration and temperature conditions. The GM Gaulin homogenizer will be used for preparation of the coal-oil mixture.

**PLANS FOR THE COMING YEAR** – The preliminary theoretical and experimental results will be extended to cover a full range of parameter values of shear rates, temperature, pressure, and concentration. Construction of the rotating rod viscometer will allow the running of tests designed to describe the rheological properties of the slurry, especially its non-Newtonian character and stability. Preparatory steps will also be taken for the next phase of investigation, which will involve characterization of atomization and spray formation in nozzles with various geometries for enhanced combustion efficiency.

## FUNDAMENTALS OF NITRIC OXIDE FORMATION IN FOSSIL FUEL COMBUSTION

WESTERN MICHIGAN UNIVERSITY

DOE - \$79,981; WMU - \$11,619

6/11/75 - 6/20/78

Principal Investigator - T. Houser

**OBJECTIVES** – This research program seeks to obtain kinetic and product distribution data for proposing a mechanism for the formation of nitric oxide from fuel-nitrogen during the combustion of coal and heavy oils. Specifically, the kinetics of the pyrolysis and oxidative pyrolysis of pyridine (because it is representative of the nitrogen-containing components of fossil fuels) will be studied. In addition, similar oxidative studies will be made on representative, condensed-ring heterocycles (e.g., quinoline, carbazole) to determine the extrapolatability of the results obtained for pyridine to more coal-like structures. The oxidation of volatile, nitrogen-containing pyridine pyrolysis products (e.g., cyanogen, HCN, acetonitrile) will also be carried out to help elucidate the mechanism of nitric oxide formation.

**RECENT WORK AND ACCOMPLISHMENTS** – The experiments using the stirred-flow reactor to obtain differential rate data were continued. The rates of disappearance of reactants were measured by using on-stream gas chromatographic (GC) and mass spectrometric (MS) techniques, while product analyses employed IR spectrophotometric as well as MS and GC monitoring. The identities of the volatile products from the inert pyrolysis of pyridine were established through comparisons of mass spectrometer cracking patterns of the product mixtures with those of known compounds. It was found that acetylene and diacetylene were important products and acetonitrile was not, contrary to previous reports. The rate of formation of HCN was determined, because it has been previously established as the most abundant pyrolysis product containing nitrogen; essentially quantitative conversion of heterocyclic nitrogen to HCN occurs at higher temperatures. Using sequential reactions, the reaction step leading to HCN formation was found to be first-order, with a frequency factor of  $10^{11.0}$  ( $\text{sec}^{-1}$ ) and an activation energy of 63.4 kcal/mole. The results of these experiments and the techniques developed lay the groundwork for the oxygen-fuel interactions. Concurrently, the oxidation of cyanogen has been studied. It was found that the only nitrogen oxide formed in the 950° to 1050°C range was  $\text{N}_2\text{O}$  in small amounts. Since no hydrogen is present, the mechanism by which NO could form may have been restricted, thus making cyanogen a poor choice for a fuel. Another interpretation could be that once the CN group has been formed through pyrolysis, the paths leading to NO formation are less likely, reducing the rate of its formation. Further oxidative studies will resolve this question.

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**PLANS FOR THE COMING YEAR** – The inert pyrolysis studies have been completed and the oxygen-fuel investigations are being initiated. It is planned to determine the conditions necessary for the formation of NO and/or NO<sub>2</sub> from heterocyclic nitrogen (pyridine) and CN nitrogen in the presence of hydrogen (acetonitrile and/or HCN). From the kinetics of fuel consumption and NO formation and the product distributions during these oxidations, it will be possible to establish the relative reactivities of these chemical structures and the extent to which pyrolysis should be carried out prior to combustion to minimize NO formation.

#### **PRE-COMBUSTION REMOVAL OF SULFUR FROM COAL BY BACTERIAL ACTION**

UNIVERSITY OF MINNESOTA  
DOE - \$40,000; University of Minnesota - \$11,534  
9/1/77 - 8/31/79  
Principal Investigator - M. Hoffman

**OBJECTIVES** – The bacterial desulfurization research program is studying microbiological processes that can be adapted for pre-combustion removal of sulfur from coal using a combination of sulfur-metabolizing and nitrogen-fixing bacteria that will effectively oxidize both inorganic and organic fractions of sulfur to water extractable compounds under ambient conditions. The results of these studies are expected to provide viable alternatives to present methods of coal desulfurization that can be implemented within a framework of existing technology and to elucidate mechanisms by which microorganisms contribute to the oxidation of metal sulfides, organic sulfides, and heterocyclic sulfur compounds.

**RECENT WORK AND ACCOMPLISHMENTS** – Representative coal samples from Ohio, Indiana, Alabama, Illinois, and Kentucky have been analyzed for sulfur content and inoculated with combinations of various bacterial strains in batch cultures. Reduction of the total sulfur content of coal samples and appearance of soluble sulfur species are monitored as a function of time. A novel technique for total sulfur determination using pyrolysis at 1400°C in the presence of hydrogen gas has been developed. Hydrogen sulfide gas generated during pyrolysis is determined gas chromatographically to give a quantitative determination of sulfur in the original sample.

**PLANS FOR THE COMING YEAR** – An automated technique for isolation and quantitative detection of soluble sulfur anions by high-speed anion-exchange chromatography (Ion-chromatography) will be adapted for monitoring sulfur conversions. The study of the dynamics of mixed-culture desulfurization will continue with emphasis on the symbiotic relationships of mutualism, commensalism, and co-metabolism. Kinetic studies will shift from batch reactors to continuous flow systems. Bacterial strains capable of degradative oxidation of thiophene derivatives will be isolated from activated sewage sludge and oil-enriched soils by trace enrichment procedures.

#### **MASS TRANSPORT CHARACTERISTICS OF ZEOLITE CRACKING CATALYSTS**

UNIVERSITY OF MISSISSIPPI  
DOE - \$235,307; UM - \$30,847  
9/30/77 - 9/30/79  
Principal Investigator - H.W. Haynes, Jr.

**OBJECTIVES** – This program intends to assess the significance of intracrystalline pore diffusion limitations when processing coal-derived syncrudes over zeolite cracking catalysts. The experimental work will involve parallel determinations of mass transport characteristics and catalyst activities

using model coal-liquid compounds. A second objective will be to test various zeolite catalysts for their ability to crack coal-derived syncrudes to naphtha. Zeolite catalysts possess selectivity and activity characteristics when processing petroleum streams that are much superior to the older amorphous silica-alumina cracking catalysts. Whether these improvements would also be realized in processing coal liquids is debatable. The large, aromatic molecules produced during coal liquefaction might have great difficulty in penetrating the zeolite crystallite. To define and assess the potential mass transport limitations in these systems is a first step towards developing catalysts specifically for refining coal-derived liquids.

**RECENT WORK AND ACCOMPLISHMENTS** — Previous work included the development and testing of a model for the application of gas chromatography to measurements of diffusion in bidisperse structured heterogeneous catalyst particles. It was demonstrated that the GC technique is ideally suited to measurements of intracrystalline zeolite diffusivities under near reaction conditions. Another project focused on the hydrogen consumption problem when processing coal derived liquids. Over a variety of catalyst types and under hydrocracking conditions, the tendency was to successively hydrogenate and crack condensed ring aromatics species at terminal rings. In the present project, these two efforts are combined into one and greater emphasis is placed upon zeolite-containing catalysts.

**PLANS FOR THE COMING YEAR** — Modifications to an existing GC-diffusivity apparatus will be completed to enable the determination of the diffusivity of condensable vapors in zeolite catalysts at elevated temperatures; the range of critical molecular diameters corresponding to the point at which the entrance to sodium zeolite Y crystallites becomes severely restricted at near reaction conditions will be identified; a simulated catalytic cracking microreactor will be constructed and debugged; and a sodium zeolite Y catalyst will be tested for its activity and selectivity in cracking model coal-liquid compounds under simulated catalytic cracking conditions. Studies will be devoted to various ion-exchanged and hydrothermally treated forms of zeolite Y. Hydrocracking studies are also planned.

## DIRECT COAL UTILIZATION USING ELECTRO-CHEMICAL DESULFURIZATION

UNIVERSITY OF MISSOURI  
DOE - \$40,000  
8/1/77 - 7/31/79  
Principal Investigator - J. Winnick

**OBJECTIVES** — An attempt is being made to develop a one-step process for removing and concentrating the  $\text{SO}_2$  and  $\text{SO}_3$  from the flue gas emanating from the direct combustion of high-sulfur fuels. The concept is based on an analogous process used in manned spacecraft for the removal and concentration of metabolic  $\text{CO}_2$ . An electrochemical cell, similar to a fuel cell, is used. The  $\text{SO}_x$  in the gas would be preferentially transferred across an electrolyte by means of an applied voltage. Depending on operating conditions,  $\text{SO}_2$  or  $\text{SO}_3$  in concentrated form would be delivered for further processing. This one-step approach avoids the reagent needs and disposal problems encountered by first generation flue-gas desulfurization techniques. It would improve on second-generation, regenerative processes in that one-step only is required, without the need for liquid contactors and pumping loops. The product could be either an  $\text{H}_2\text{-SO}_2$  mixture or  $\text{SO}_3$  for the manufacture of concentrated ( $>100$  percent) sulfuric acid.

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**RECENT WORK AND ACCOMPLISHMENTS** – Tests with an aqueous electrolyte showed that the bisulfite ion was too unstable to be transported without undergoing oxidation or reduction. As a result, tests were initiated using nonaqueous electrolytes, the first of which was the eutectic (Li/K/Na) sulfate. Very promising results were obtained; nearly complete removal of the  $\text{SO}_2$  was effected from synthetic flue gas. Concentrated  $\text{SO}_3$  evolved from the recovery chamber.

**PLANS FOR THE COMING YEAR** – First, the best electrolyte must be found. Several non-aqueous mixtures are under investigation. Second, several electrode materials are being tested. Third, a new cell design is being constructed, modeled after successful high-temperature fuel cells. Another area for exploration involves the use of hydrogen as a reducing gas in the recovery chamber since the emitted gas would be a mixture of  $\text{H}_2$  and  $\text{SO}_2$ . This mixture is easily treated by existing technology to sulfur and water.

## CHEMICAL AND PHYSICAL STABILITY OF REFRACTORIES IN COAL GASIFICATION

UNIVERSITY OF MISSOURI-ROLLA  
DOE - \$121,385; UM-Rolla - \$10,349  
5/1/76 - 4/30/78  
Principal Investigator - D.E. Day

**OBJECTIVES** – The exterior metal shell of a coal gasification vessel is lined with a thermally insulating refractory to prevent the shell from overheating and coming into direct contact with hot corrosive gases ( $\text{CO}$ ,  $\text{H}_2$ , steam, etc.). At elevated temperatures, highly erosive conditions occur as a result of the movement of solid coal particles, ash, and molten slag. These conditions necessitate the development of refractories that can withstand the chemical corrosion and mechanical erosion existing in coal gasifiers for long periods of time. This investigation is establishing the chemical corrosion resistance of refractories exposed to high-pressure/temperature gas and liquid environments encountered in coal gasification. Specific objectives are to achieve an understanding of chemical reactions that may occur in refractories exposed to conditions representative of those at the cold face of refractory lining in gasifiers, assess the relative importance of these reactions to those physical/chemical properties needed for long service-life, and identify those refractory systems providing optimum service performance, particularly in regard to the refractory bond phases.

**RECENT WORK AND ACCOMPLISHMENTS** – Sixteen commercial and laboratory prepared calcium aluminate cement-bonded and phosphate-bonded refractories have been tested in the DOE in various  $\text{CO}$ -steam atmospheres with and without  $\text{H}_2\text{S}$ . Refractory specimens have been exposed to both saturated and unsaturated atmospheres for 30 days at temperatures and pressures of up to  $1000^\circ\text{F}$  and 1000 psia, respectively. The cement bonded castables, especially the high and intermediate alumina, showed the greatest evidence of chemical reaction with the test atmospheres. Up to  $532^\circ\text{F}$ , the principal reactions were the hydration of  $\text{Al}_2\text{O}_3$  to form boehmite ( $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ) and the reaction of the  $\text{CaO}$  component of the cement with  $\text{CO}$  to form  $\text{CaCO}_3$ ; at higher temperature ( $1000^\circ\text{F}$ ), only  $\text{CaCO}_3$  formed. The two to threefold increase in flexural strength observed when these castables were exposed to saturated atmospheres was essentially proportional to the quantity of boehmite formed during exposure. The  $\text{AlPO}_4$  bond phase in the phosphate-bonded refractories, fired to  $1000^\circ\text{F}$  prior to exposure, showed no significant chemical reaction with the test atmospheres except for saturated conditions. In the saturated atmospheres, leaching of the phosphate bond from the refractory was indicated by deposits of hydroxyl apatite,

$\text{Ca}_5(\text{PO}_4)_3\text{OH}$ , found in the vessel after exposure. The flexural strengths of these specimens were reduced by approximately 50 percent. The most chemically aggressive gases present in the atmosphere are CO and  $\text{H}_2\text{O}$  (steam). No major effect upon the chemical reactions occurring in the refractories or upon their flexural strength was found at 1 volume percent  $\text{H}_2\text{S}$  in the atmosphere. The degree of saturation of the test atmosphere with steam is one of the most important factors affecting the performance of the refractories.

**PLANS FOR THE COMING YEAR** – Work will include exposure tests in CO-steam atmospheres containing 1 volume percent  $\text{H}_2\text{S}$  for CO/ $\text{H}_2\text{O}$  ratios = 0.1, 1, and 3; continuation of XRD and DTA/TGA analyses; and a long-term exposure of 60 days in the DOE atmosphere with 1 volume percent  $\text{H}_2\text{S}$ .

### MECHANISMS AND RATE OF ALKALI TRANSPORT IN TYPICAL COAL GASIFICATION PROCESSES

UNIVERSITY OF MISSOURI-ROLLA

DOE - \$153,127

9/76 - Continuing

Principal Investigator - G. Lewis

**OBJECTIVES** – The degree and significance of alkali vaporization and transport is to be evaluated and assessed under the temperature, pressure, and atmospheric conditions characteristic of various coal gasification processes. The work will involve both calculated relations and experimental confirmation.

**RECENT WORK AND ACCOMPLISHMENTS** – The thermodynamic stability of the alkali-oxides, -sulfates, nitrates, -carbonates, -sulfides, -cyanides, and selected silicates at various temperatures, pressures, and atmosphere compositions (assumed to exist in high- and low-Btu gasifiers) has been evaluated by calculation. These calculations are being reworked into a graphical form to make relations involving significant alkali transport more easily identified and compared to other reactions.

Thermal analysis (weight loss) experiments using the “ERDA atmosphere” have been made on selected alkali compounds in a Mettler Thermoanalyzer. Significant modifications to the apparatus have been made to allow operation under reducing conditions. The experiments are presently limited by severe deterioration of the fused silica Mettler furnace after alkali transport has been observed. A thermal transport furnace system (operated at atmospheric pressure) has been constructed to avoid the materials problems involved in the Mettler system and it is undergoing initial performance testing. The high-pressure thermal transport furnace has been assembled, and preliminary testing is being started. The high-pressure system should be fully operational within 1 month.

**PLANS FOR THE COMING YEAR** – The Mettler thermal analysis experiments have indicated that the sulfates of potassium and sodium are significantly transported by the “ERDA atmosphere,” in addition, those compounds tend to migrate by a creep or diffusional mode from the crucible containing them. This migration occurs even when a lid is present on the crucible; therefore, these two compounds will be the first tested in the transport systems. Identification of the major gaseous species will be sought in the transport experiments also. Because creep of the alkali-rich residue to cooler regions may be a more significant transportation mechanism than vapor transport, this



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problem will be investigated along with the vaporization. High-pressure transport experiments will be carried out on the other compounds cited and correlated with the calculated results.

## CATALYTIC HYDROGENATION OF COAL-DERIVED LIQUIDS

MONTANA STATE UNIVERSITY, BOZEMAN

DOE - \$155,932; MSU - \$24,544

6/20/75 - 6/19/78

Principal Investigators - L. Berg, F.P. McCandless

**OBJECTIVES** – This project seeks to develop processes to convert liquefied coal into clean distillate fuels at the lowest possible cost. The program is limited to research on liquefied coal made by the COED, SRC, Synthoil, and H-Coal processes, all of which have reached the pilot plant scale. Either existing or new hydrotreating catalysts will be used for the hydrocracking of the liquefied coal at conditions of temperature, pressure, residence time, and hydrogen flow rate that are relatively moderate.

**RECENT WORK AND ACCOMPLISHMENTS** – COED pyrolysis oil has been hydrotreated in a continuous trickle flow reactor at 415°C, 800 psig, and a hydrogen flow rate of 10,000 scf/bbl. Six cobalt molybdate, five nickel tungsten, and five nickel molybdate catalysts of various manufacture have been tested at the above conditions. It appears that the nickel and cobalt molybdate catalysts have similar hydrotreating activity, the cobalt molybdate removing more sulfur while the nickel molybdate removes more nitrogen. The nickel tungsten catalysts showed much lower activity for hydrotreating than the catalysts containing molybdenum. The best commercial catalysts from an overall point of view seem to be the nickel molybdates Cyanamid HDS9A and Harshaw HT500. They removed the most nitrogen, which is the bottleneck in hydrotreating this feed. Also, from the limited pore size data available, these tests show that nitrogen removal, and to a lesser extent sulfur removal, is very much limited by pore diffusion. Five catalysts have been tested that were made at MSU: cobalt, nickel, and molybdenum on various Norton and Ketjen supports. The results of the tests have shown that pore size, silica content, and molybdenum content greatly affect hydro-treating ability. On SRC, 40 different catalysts have been tested in 44 batch runs and 40 continuous fixed-bed reactor runs. The best catalysts include a cobalt-nickel-molybdate made at MSU. The best hydrocracking results have yielded a product with 15 volume percent gasoline boiling range material, 46 volume percent diesel range material, and 27 volume percent gas oil range material. The best nitrogen removal has been 65 percent, and the best sulfur removal has been 72 percent. Synthoil has been hydrotreated in a continuous trickle flow reactor at 450°C, 800 psig and a hydrogen flow rate of 10,000 scf/bbl. The liquid flow rate was varied to give liquid hourly space velocities ranging from 1 to 2. In batch runs, 27 catalysts have been screened; and 12 catalysts have been tested in the continuous reactor, including 3 cobalt molybdate, 4 nickel molybdate, and 3 nickel tungsten. The cobalt molybdate and nickel molybdate catalysts are more effective in hydro-treating Synthoil than the nickel tungsten catalysts. The best nitrogen removal and hydrocracking was achieved by Shell catalyst 324, and the best sulfur removal was accomplished with Harshaw HT500 catalyst. Three MSU catalysts have been tested in batch runs and one of these, a cobalt-nickel-molybdenum-iron catalyst, gave the best hydrocracking of all of those so far tested.

**PLANS FOR THE COMING YEAR** – The best commercial catalysts for denitrogenation, desulfurization, and hydrocracking of the liquefied coals have been determined. Major emphasis will be on the fabrication and testing of new catalysts composed of combinations of cobalt, nickel, tungsten,



and molybdenum on supports having specific characteristics of chemical composition, surface area, pore volume, and pore diameter.

## DIRECT CONVERSION OF LIGNITE TO CHEMICAL FEEDSTOCKS

MONTANA STATE UNIVERSITY

DOE - \$19,900

9/1/77 - 8/31/78

Principal Investigator - W. Scarrah

**OBJECTIVES** – This program seeks to: (1) determine the potential for directly converting lignite to chemical feedstocks via a process combining technologies from molten salt catalysis and solvent refining; (2) investigate the effects of alkali metal halides added to zinc chloride catalyst on product distribution and hydrocarbons-salt separation; (3) determine the effects on product distribution of hydrogen sources as influenced by reducing gas composition, lignite moisture, and added water; and (4) identify the significant processing variables (alkali metal halides and hydrogen sources) preparatory to more comprehensive process studies.

**RECENT WORK AND ACCOMPLISHMENTS** – This work is in its initial design stages.

**PLANS FOR THE COMING YEAR** – A fractional factorial experimental design will be used with the alkali metals lithium, sodium, and potassium to evaluate halides (chloride, bromide, iodide), reducing gases (hydrogen, carbon monoxide, syngas), and water sources (wet lignite, dry lignite, dry lignite plus added water). Batch runs using a rocking autoclave will be appropriate for undergraduate student operation because of their simplicity and shortness. Product analyses will include gas chromatography and nuclear magnetic resonance.

## ENVIRONMENTAL EFFECTS FROM LEACHING OF COAL CONVERSION BYPRODUCTS

UNIVERSITY OF MONTANA

DOE - \$74,948; UM - \$17,400

6/75 - 6/78

Principal Investigators - W.P. Van Meter, D.E. Erickson

**OBJECTIVES** – Most of the trace mineral content of coal will remain with the solid residues or byproducts of a gasification facility. Surface landfill or strip-mine backfill are among the more attractive disposal options for this material. Either would subject the material to probable percolation or infusion by precipitation or groundwater. This project will study solid byproducts from existing pilot plant or full-scale facilities by column leaching methods with the purpose of determining whether potentially hazardous amounts of toxic elements may be leached into ground or surface water. This information will provide a basis for decisions concerning the ultimate disposal of wastes from coal conversion plants.

**RECENT WORK AND ACCOMPLISHMENTS** – Leaching procedures and all the associated chemical analyses have been completed for 23 column leaching runs. These tests involve 12 specimens from six different coal conversion facilities; 9 other column runs are complete with some analytical work remaining. A specimen of residue material was received from the Westinghouse low-Btu gasifier in Pittsburgh late in the summer. Plans for this past year had included testing of materials from Bituminous Coal Research, Inc. and the Bi-Gas high-Btu gasifier, but specimens have

not been received. Results are expressed in two ways. In some cases, column effluent solutions have carried metal concentrations in excess of the limits prescribed by the EPA for domestic water supplies. Manganese, nickel and mercury were found in the effluents from the Leatherhead, England COED process; the Sasol, South Africa Lurgi plant; the CO<sub>2</sub> Acceptor process; and the MERC stirred-bed process at levels from fivefold to a thousandfold higher than the EPA limits (maximum observed concentrations of short-time duration). In another format, the integrated amount of each individual element has been tabulated as a percentage of the amount of residue material from which it was leached. Most of these values are quite small (0.01 to 10<sup>-6</sup> percent) for those elements commonly perceived as toxicity hazards, but 1 year's output of waste from a full-scale plant (250 x 10<sup>-6</sup> ft<sup>3</sup>/day) will contain 100 pounds of leachable mercury. Some of the more common elements appear in much larger amounts. They are found in natural waters but at higher levels cause the problem of brackishness. MERC and Synthane PDU materials contain between 0.5 and 1.0 percent leachable calcium. Material from the Sasol plant is over 2 percent leachable sodium.

**PLANS FOR THE COMING YEAR** – The schedule of column and analytical operations will allow for completion by spring 1978 of testing residue materials now on hand. Several of the sites represented produce materials that do not closely resemble what the proposed full-scale facility will produce. Efforts are continuing to obtain specimens more accurately representative of full-scale conditions and more closely coordinated with meaningful pilot plant operating parameters.

## STUDY OF FLY-ASH FORMATION MECHANISM

UNIVERSITY OF NEW HAMPSHIRE  
DOE - \$223,740; UNH - \$4292  
2/76 - 2/79  
Principal Investigator - G.D. Ulrich

**OBJECTIVES** – Recent studies of fly-ash emitted from stationary combustion equipment suggest that substantial numbers of sub-micron particles are discharged even from units having electrostatic precipitators. This fraction is the size which is least efficiently collected and most damaging physiologically. With the prominence of coal as a fuel, pollution from sub-micron fly-ash particles will receive increasing attention. Work in this laboratory falls within two categories. The first is a fundamental investigation of quench temperature, sticking coefficient, aggregate morphology, particle-size distribution, electrical charge effects, and other phenomena that influence the formation and growth of pure and mixed metal oxide particles. This study is being conducted with the aid of a precision laboratory burner. The second category involving growth of ash particles in a full-scale commercial utility boiler is an effort to determine fly-ash characteristics as a function of temperature and residence time in the boiler. Two cyclone-fired units at a nearby power plant have been made accessible for this work. With results from the fundamental study, a model will be formulated to describe particle behavior in commercial boilers.

**RECENT WORK AND ACCOMPLISHMENTS** – Preliminary sampling, conducted at the Merrimack No. 2 Boiler (Public Service Company of New Hampshire), confirms that there are two types of ash generated in a coal-burning unit: entrainment or residual ash that remains after the complete combustion of an entrained coal particle, and condensation ash produced from the oxidation and precipitation of volatilized metallic species. The entrainment ash passes through the boiler largely unchanged and consists typically of 2 to 50 micron, spherical, non-porous, discrete particles. Condensation ash, on the other hand, appears to precipitate as a result of rapid quenching during

passage through the tube banks. The particles, invisible in the optical microscope, appear under the electron microscope as clumps or aggregates of sub-micron fused oxide droplets. Though representing less than 1 percent of the fly-ash mass, the sub-micron fraction amounts to more than 99 percent of the total ash on a number basis. In contrast to suggestions by others that enrichment of some metals among the smaller particles is caused by surface condensation, enrichment is caused apparently by condensation ash which precipitates directly from the gas phase and is disproportionately collected on entrainment-ash particles according to surface area. Preliminary analysis isolates the vaporization of volatile ash-containing species in the reducing region of a burning coal particle as a logical source of entrainment ash. In laboratory studies, the processes influencing growth of sub-micron silica particles have been studied using a premixed flat-flame burner. Results indicate two important growth-controlling factors. One is the collision rate between particles which is accurately described by Brownian theory. The second is fusion rate which is strongly dependent on viscosity and on temperature. The flocculated morphology of the sub-micron particles is a logical consequence of simultaneous collisional and coalescence control of particle growth.

**PLANS FOR THE COMING YEAR** — Laboratory studies will move from pure oxides such as titania and alumina to oxide mixtures resembling those found in coal ash. Continued boiler sampling is planned. These power-plant results will be combined with equilibrium calculations and laboratory particle-growth studies to develop a general fly-ash formation mechanism.

## OPTIMIZATION OF PYROLYTIC CONVERSION OF COAL TO CLEAN FUEL

PRINCETON UNIVERSITY  
DOE - \$341,931; PU - \$20,724  
1/1/76 - Continuing  
Principal Investigator - M. Summerfield

**OBJECTIVES** — This program seeks to: (1) develop a mathematical model to represent the important steps in the coal pyrolysis process; (2) apply the overall devolatilization scheme to reactors of several classes, specifically, fixed-bed, fluidized-bed, entrained flow, and multi-state fixed and fluidized beds, in connection with overall optimization of the coal conversion to clean fuel; (3) couple knowledge of the pyrolysis steps with subsequent steps of a coal conversion process that combine to produce a preferred fuel. These steps would include hydrotreating the oils to remove sulfur and produce clean liquid fuels and hydrogenation of the gaseous fractions and other upgrading reactions to produce pipeline gas of 900 to 1000 Btu/scf, hhv.

**RECENT WORK AND ACCOMPLISHMENTS** — A computer program was developed for kinetic analysis of single- and multiple-step coal pyrolysis mechanisms and was applied to thermogravimetric (TG) data for Wyodak coal. The Reidelbach-Summerfield (R-S) coal pyrolysis model fit the data better than a single-step mechanism, although further investigations in this area are needed. A second computer program was developed for obtaining kinetic parameters from TG obtained at several linear rates of temperature rise and was successfully applied to Wyodak coal. Other programs were developed to digitize graphed TG data by a magnetic reader, to transfer these data to provide smoothed first derivatives. A computer program was developed to combine TG and differential scanning calorimetry (DSC) data for coal to provide better understanding of the mechanism and energetics of pyrolysis and to yield kinetic equations and parameters that are more accurate and representative of that mechanism.

For Task 2, the Chemical Data Systems (CDS) pyrolysis/gas chromatography (GC)/peak identifier system was installed. A significant effort was required to determine the most effective way to use this instrumentation. Tiny samples of four different coals were pyrolyzed under similar conditions, and GC results were obtained for each. Comparison showed qualitative similarities but quantitative differences. Known samples were compared to pyrolysis results, and some probable products were identified. A quartz tube fixed-bed reactor was fabricated to aid in the development of a more elaborate metal reactor. Montana lignite samples were pyrolyzed in flowing helium atmospheres using heating rates of 5° and 10°C/min., and uncontrolled (rapid) heating. Pressure drop and sample temperature were monitored; two thermocouples were placed at different radial locations to determine temperature gradients. Gases were sampled for the slower runs, after the products passed through a carbon dioxide ice-cooled trap and were analyzed by GC. Yield vs temperature curves were in good agreement with literature values for Wyodak coal. Most of the components of the metal reactor have been designed, and parts and materials are currently being procured for their fabrication. Many sections of the shell and tube entrained flow reactor were designed and fabricated from Hastelloy B, and this apparatus is being assembled. A belt-type coal feeder was designed and fabricated. Incoloy 800H has been ordered as a backup material for Hastelloy, for it is expected to have much better sulfur corrosion properties.

For Task 3 a computer program for modeling the fixed-bed reactor has been completed and is being tested. Preparation of a computer program for fluidized-bed pyrolysis reactors is envisioned after completion of a literature review. The solutions of the fixed-bed reactor model are expected to help determine some design features of the metal reactor.

**PLANS FOR THE COMING YEAR** – Run TG and DSC tests for specific coals as tested in reactors and evaluate kinetic parameters for inclusion in reactor modeling programs. Run CDS pyrolyses and product analyses for specific coals to be tested in reactors to establish GC routines. Complete fabrication of metal fixed-bed and entrained-flow reactors. Perform coal pyrolysis tests, as required, to determine effect of various operating parameters on yields of various products. Analyze data with aid of reactor modeling programs to quantify primary and secondary reaction kinetics. Complete development of reactor modeling computer programs for various reactors, using kinetic and pyrolysis reactor data, and extend technology to listed reactors to determine overall optimization of conversion of coal to clean fuel. Carry out TG, DCS, CDS, and pyrolysis tests in hydrogen atmospheres. Include hydrogenation in reactor models and develop required computer programs. Carry out optimization calculations for hydrogasification reactors.

## DESULFURIZATION WITH TRANSITION METAL CATALYSTS

STATE UNIVERSITY OF NEW YORK AT BINGHAMTON

DOE - \$39,932

8/1/77 - Continuing

Principal Investigator - J.J. Eisch

**OBJECTIVES** – This study evaluates the mechanism and efficiency of novel, homogeneous, transition-metal desulfurizing agents for organosulfur compounds. Because the molecular structures present in native coal contain organosulfur subunits, efficient, homogeneous desulfurizing agents may be useful in studies of the structure of coal itself. Moreover, these same agents, if active catalytically, would be valuable in freeing fuels or organic chemicals obtained by coal pyrolysis from the contaminating organosulfur components.

**RECENT WORK AND ACCOMPLISHMENTS** – Sulfur heterocycles found in certain native coals and typical of the stable organosulfur compounds found in coal-derived fuels were chosen as test substrates for the desulfurizing efficacy of nickel (0) complexes. Preliminary work has already shown that although bis-1,5-cyclooctadiene-nickel(0) is unreactive, a 1:1 admixture of this nickel reagent with  $\alpha, \alpha'$ -bipyridyl provides an effective desulfurizing reagent (conditions: homogeneous solution in tetrahydrofuran in the range of 40° to 60°C). Heterocycles undergo desulfurization with ring contraction. The sulfur removed forms NiS, which upon workup with aqueous acids yields H<sub>2</sub>S. The behavior of dibenzothiophene, on the other hand, seemed to be different; treatment with the nickel(0) reagent and subsequent hydrolysis with HCl yielded biphenyl. Now that a useful stoichiometric desulfurization agent has been found for homogeneous, mild conditions, catalytic activity of such nickel(0) systems is being investigated under a hydrogen gas atmosphere and with various hydrogen sources.

**PLANS FOR THE COMING YEAR** – Besides an investigation of the catalytic activity of Ni(0) complexes under a hydrogen atmosphere, a study of the desulfurizing action of nickel(0) complexes on mercaptans (R-SH), disulfides (R-S-S-R) and diaryl sulfides (Ar-S-Ar) will be undertaken. Because organic sulfur in motor fuels derived from coal occurs in these forms, it is important that effective desulfurizing conditions be found for these compounds. Not only will the efficacy of the aforementioned nickel(0) reagents be examined for these compounds, but the value of some recently discovered titanium reagents will also be examined.

#### USING COAL/WATER/OIL EMULSION AS CLEAN LIQUID FUEL

ADELPHI UNIVERSITY  
DOE - \$99,722; AU - \$14,308  
7/1/77 - 6/30/78  
Principal Investigator - J. Doohar

**OBJECTIVES** – Studies have shown that coal/water/oil emulsions can be burned in a conventional oil-fired furnace; however, the behavior of the emulsions under pumping and atomization and their ultimate value as fuels have been shown to be strongly dependent on the coal used in the emulsion. This work will correlate coal properties with emulsion properties, especially those parameters relevant to practical coal utilization for emulsion fuels.

**RECENT WORK AND ACCOMPLISHMENTS** – An extensive testing program is being carried out to evaluate the properties of coal that influence shelf-life stability and stability under the shear rates encountered in typical pumping setups. It has been demonstrated that coal/water/No. 2 oil emulsions can be burned in modified oil furnaces with the same efficacy as straight oil. The emulsions show thixotropic behavior both when tested in the viscometer and when pumped through a section of the pipe. The flow and combustion properties of the emulsions are strongly dependent on the type of coal used.

**PLANS FOR THE COMING YEAR** – The emulsions and slurries that show good stability will be burned in a full-size working boiler and evaluated for combustion and thermal efficiencies, ash deposition, and radiation. Limestone or soda ash will be added to the emulsions for removing SO<sub>2</sub>. The NO<sub>x</sub> levels of emulsions and coal/oil slurries will be compared. Plans are to use one of the Adelphi University boilers that is currently burning oil. The boiler will be completely instrumented to monitor all important parameters. Studies will be done on the effect of particle-size distribution

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and concentration of coal and water in the emulsions on the radiation and rheology of the emulsion.

## IMPROVED TECHNIQUES FOR GASIFYING COAL

THE CITY COLLEGE OF THE CITY UNIVERSITY OF NEW YORK

DOE - \$471,000; CCNY - \$219,200

6/30/76 - 1/30/78

Principal Investigators - R.A. Graff, J. Yerushalmi

**OBJECTIVES** — This program intends to lay the groundwork for a development by industry of new techniques for converting coal into gaseous and liquid products. The program explores the underlying features and operating characteristics of beds of fine solids fluidized at high velocities, especially the fast fluidized bed; the flash heating of raw coals in gases containing hydrogen at elevated pressures with control of the residence time of the vapor; and the flash heating of raw coals in steam at high pressure.

**RECENT WORK AND ACCOMPLISHMENTS** — A laboratory reactor system has been developed for the determination of products obtainable from the flash heating of raw coal in flowing hydrogen at pressures up to 100 atm. The system provides for independent control of three important time parameters: heating rate, solids-contact time, and vapor-product residence time. A series of runs has been completed in a variety of coals, representing a cross-section of U.S. coals. Comparative analysis of BTX yields is underway. A brief study of yields obtainable at short residence time (0.18 sec) and high temperature (up to 955°C) reveals that at 955°C the total liquid yield is high (over 40 percent carbon conversion) but low in BTX (4 percent carbon conversion). Tests with lignite show no effect of drying on yield structure. Development of gas chromatography for analysis of heavy products is continuing. A system to study the effects of the mixture of hydrogen and steam on reaction products is also under development. Both the 6-in. and two-dimensional fast fluidized beds have undergone extensive modifications. A new solids transfer line has been installed in the 6-in. system, with the aim of studying the effect of partitioning the fluidizing gas between injection locations at various proportions upon the operation of the fast bed, and specifically upon the extent of bypassing of gas from the fast to the slow bed. Study of the behavior of several solids in the fast-fluidized-bed regime is underway as well as the modes of transfer of solid from a standpipe to the bottom of a circulating high-velocity fluidized bed. Results of gas backmixing experiments and the efficiency of cyclones for separating solids and gases as well as data on turbulent and bubbling regimes have been obtained.

**PLANS FOR THE COMING YEAR** — Work on the flash hydrogenation project will continue in the exploration of yields of BTX from a variety of coals, representing a cross section of U.S. coals. Modifications on the configuration of the fast beds will continue, including the study of a lateral transfer line connecting the slow and fast bed. Testing with different solids will also continue.

## SIMPLEX PROCESS FOR GASIFICATION OF COAL AND MUNICIPAL SOLID WASTE

COLUMBIA UNIVERSITY

DOE - \$111,000

4/1/77 - 3/31/78

Principal Investigator - H.W. Schulz

**OBJECTIVES** — This program seeks to develop a slagging moving-burden gasifier of high productivity capable of processing caking coals and coal fines. A key feature of the Simplex process is the compacting or briquetting of the burden that comprises a well-blended mixture of an Eastern bituminous caking coal encapsulated in shredded municipal solid waste (MSW), biomass, or lignite. The effect of the principal briquette formulation and fabrication parameters on the structural integrity and caking propensity of preformed Simplex briquettes in the drying, pyrolysis, and reaction zones of the gasifier is being defined. The parameters being evaluated include: type and particle size of coal, form of MSW or refuse-derived fuel, the coal/MSW ratio, choice of low-cost binder (if any), compaction pressure, and briquette size. Another specific objective is to determine heat transfer, drying, and relative reaction rates of Simplex briquettes at temperature levels from 300° to 3000°F.

**RECENT WORK AND ACCOMPLISHMENTS** — Employing highly caking eastern coals (with a free-swelling index of 8.5) and shredded air-classified MSW, the absence of swelling, tar exudation, and adhesion was demonstrated at coal/MSW ratios as high as 3:1 when stacks of cylindrical Simplex briquettes were pyrolyzed in an inert atmosphere over the temperature range from 600° to 1400°F. Test procedures were developed to measure the load-bearing capacity and friability of Simplex briquettes over a broad range of compositions in three states: as pressed, after drying at 250°F, and after being coked in a stream of nitrogen at 1900°F. Satisfactory briquette strength was realized without the use of added binder at a compaction pressure of 5000 psi. Black liquor (from the paper industry), black strap molasses, and waste starch were found to be effective binders and produced structurally sound briquettes at compaction pressures as low as 2000 psi for coal/MSW ratios from 0.7:1 to 3.5:1. Dewatered sewage sludge was shown to be a binder of intermediate effectiveness. The binder properties of tar oils recovered from the process are still to be evaluated. In the course of the program, 1500 Simplex briquettes of adequate structural integrity and non-agglomerating properties were produced from nine different coals, three commercially available forms of MSW, and five low-cost binders; they were fabricated as 2- and 3-in.-diameter cylinders by ram compaction and quantitatively evaluated for crushing strength, penetrability, and friability under tumbling. As a result, a commercially attractive range of briquette fabrication parameters has been defined.

**PLANS FOR THE COMING YEAR** — Some additional screening tests are planned to investigate several other coal types (including anthracite and lignite); other forms of biomass to serve as the encapsulating matrix; the use of recycled tar oils in the briquette formulation; and the drying properties of larger diameter briquettes. In collaboration with a manufacturer of briquetting machines, the adaptability will be evaluated of the high-speed rotary press to produce pillow-shaped briquettes 4 to 6 in. in length to serve as feedstock for pilot plant development of the Simplex process. It is projected that the pilot plant tests will be carried out initially in the 16-in.-diameter nonslagging atmospheric-pressure gasifier at the Morgantown Energy Research Center to be followed by a pilot plant demonstration of the process in the 25-t/d slagging medium-pressure gasifier at the Grand Forks Energy Research Center.

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## FLUID DYNAMICS OF FLUIDIZED BED PACKED WITH HEAT EXCHANGERS

NEW YORK UNIVERSITY  
DOE - \$486,415; NYU - \$25,000  
2/1/76 - 1/31/78  
Principal Investigators - V. Zakkay, G. Miller

**OBJECTIVES** – Approaches for using coal to produce electricity include the facility that has been built by Pope, Evans, and Robbins at Rivesville, West Virginia and the pilot plant at the Exxon Research and Engineering Company. The bed at Rivesville operates at atmospheric pressure and utilizes a horizontal tube concept based on standard boiler designs. This research is performed in support of the Rivesville program as well as in support of pressurized fluidized beds, such as the Exxon and Curtiss Wright designs. The research provides information on efficient methods of extracting heat from fluidized beds.

**RECENT WORK AND ACCOMPLISHMENTS** – Both the 1- and 3-ft-diameter fluidized beds have been completed. Research has been focused on the determination of heat transfer coefficients in the 1-ft fluidized bed over a wide range of operating conditions. Experiments were conducted by utilizing a vertical coil arrangement similar to the one used at Exxon and a horizontal coil arrangement similar to the one used by Pope, Evans, and Robbins. The results indicate that the heat transfer coefficient is significantly affected by the coil configuration, velocity profile, superficial velocity, and pressure. All the tests thus far have been conducted with particular sizes of 1/16-inch to 1/8-inch.

**PLANS FOR THE COMING YEAR** – Tests are presently being conducted in the 3-ft-diameter bed to provide information on the effect of scale on the differences in film coefficient in deep beds and shallow beds, and to investigate various geometries of heat exchangers.

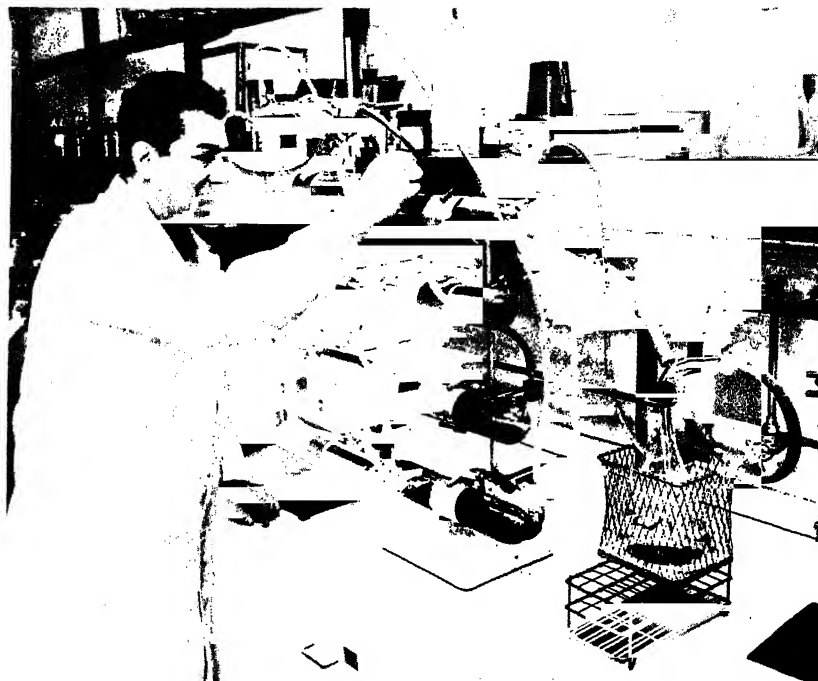
## OPTIMIZATION OF BACTERIAL LEACHING FOR REMOVAL OF PYRITE FROM COAL

STATE UNIVERSITY OF NEW YORK  
DOE - \$19,500; SUNY - \$2400  
8/1/77 - 9/31/78  
Principal Investigator - N. Lazaroff

**OBJECTIVES** – This research seeks to test and improve laboratory-scale bacterial leaching systems that may be used to develop industrial-scale removal of pyritic sulfur from aqueous slurries of crushed coal. Concern over the environmental impact of increased use of coal has motivated efforts to obtain new sources of low sulfur coal and to utilize processes of coal conversion that will minimize sulfuric emission. The focus of this research is to assess the rate limiting processes for the solubilization of pyrite in coal that occur during the recirculation of lixiviants containing iron oxidizing Thiobacilli. The parameters under consideration are the relationship of bacterial population, anionic composition, iron content, and oxygen concentration of the leach water systems to rate of pyrite solubilization in coals.

**RECENT WORK AND ACCOMPLISHMENTS** – A new type of experimental laboratory leaching system has been developed that simulates the variable flow, multistage heterogeneous system of lixiviant and coal that is obtained if minewater is recirculated over crushed coal and associated minerals in a mine. The gas, liquid, and solid phases can be sampled conveniently without disruption of the leaching process. Preliminary analyses of the experimental leaching systems are in progress.





*Nine-Unit Battery of Coal Leachers*

**PLANS FOR THE COMING YEAR** – Operating, sampling, and analysis of multiple leaching systems will be performed to enable design of a pilot plant bacterial leaching system for solubilization of pyrite in coal.

#### **NITROGEN OXIDE PRODUCTION FROM CHEMICALLY BOUND NITROGEN IN COAL**

STATE UNIVERSITY OF NEW YORK AT BUFFALO

DOE - \$38,900

9/1/77 - 8/31/80

Principal Investigator - L.A. Kennedy

**OBJECTIVES** – This program is designed to assess the relative importance of factors that influence the conversion of chemically bound nitrogen in coal to nitrogen oxides during combustion. Work will focus on the role of flame temperature, coal and char composition, and aerodynamic mixing.

**RECENT WORK AND ACCOMPLISHMENTS** – This report covers the period September 1, 1977 to September 31, 1977. The initial month's work was directed towards organizing this project both in terms of personnel and equipment. Scientific and technical training was directed toward students in the Mechanical Engineering Department at SUNY. The initial design of the combustor was evaluated and discussions with vendors for its fabrication were begun.

**PLANS FOR THE COMING YEAR** – During the period October 1, 1977 to February 28, 1978 the final design of the combustor to be used in the experiments will be completed and sent out for fabrication. Orders will also be placed for NO<sub>x</sub> emission equipment which is the cost sharing contribution of SUNY.

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## NOVEL CATALYST SUPPORTS FOR HYDRODESULFURIZATION OF COAL

STATE UNIVERSITY OF NEW YORK AT BUFFALO

DOE - \$243,553; SUNY - \$56,187

6/3/75 - 6/2/78

Principal Investigator - S.W. Weller

**OBJECTIVES** – This program investigates: coal hydrodesulfurization catalysts of varying but controlled pore size of support, in both pelleted and monolith configuration; physicochemical methods for characterizing the specific surface area of the supported transition metal oxides or sulfides, for application to fresh and deactivated catalysts; and catalytic activity in model reactions.

**RECENT WORK AND ACCOMPLISHMENTS** – The effects of stirring rate, catalyst pore size, and catalyst presulfiding on the hydrodesulfurization of a West Virginia coal (4.3 percent S) have been studied in a new Magnedrive autoclave, with tetralin as solvent. With unsulfided particulate catalyst, 200Å pores gave highest oil yield and hydrogen consumption. With presulfided catalyst, highest oil yield and hydrogen consumption were obtained with 100Å. Sulfur contents were about 0.7 percent for the product asphaltenes and 0.3 percent for the oils. Monolith catalysts of three different configurations have also been tested in the Magnedrive autoclave. Highest oil yields and highest hydrogen consumption occurred with that monolith that had 200 square holes/in.<sup>2</sup>. This catalyst produced asphaltenes of ~ 0.6 percent S and oils of 0.2 percent S. The hydrogenolysis of methyl naphthalene, a good model compound, has been studied at 450° and 500°C, in the absence of catalyst and in the presence of Harshaw Co/Mo/Al<sub>2</sub>O<sub>3</sub> catalyst. After 1 hour at 450°C in the absence of catalyst, no hydrogen consumption or methane production occurs. Catalyst at 450°C results in substantial hydrogen consumption, presumably to give methyl tetralin, but no methane production. Hydromethylation is rapid, however, in the presence of catalyst at 500°C; methane and naphthalene are major products. A laboratory method has been developed for making Mo/Al<sub>2</sub>O<sub>3</sub> catalysts in which the molybdena is preferentially distributed in a relatively thin shell near the external surface of the pelleted catalyst. The method involves impregnation of alumina pellets with an aqueous solution of a molybdenum compound, heat treatment and reduction either with a hydrazine solution or with flowing hydrogen. Preliminary results have been obtained on the kinetics and mechanism of the vapor-phase hydrodenitrogenation of pyrrolidine over Co/Mo/Al<sub>2</sub>O<sub>3</sub>.

**PLANS FOR THE COMING YEAR** – The autoclave studies on West Virginia coal, with both particulate and monolith catalysts, will be extended to the use of methyl naphthalene as solvent. The “shell” catalysts of Mo/Al<sub>2</sub>O<sub>3</sub> will be characterized both with respect to specific surface area of the molybdena and for comparative catalytic activity in model test reactions. The thermal deactivation of “shell” pelleted catalyst and of monolith catalyst will be studied as a means of determining the importance of thermal deactivation in the aging of Synthoil catalyst.

## DETONATIVE COAL GASIFICATION

NORTH CAROLINA STATE UNIVERSITY

DOE - \$19,800

9/1/77 - 8/31/78

Principal Investigator - T.H. Pierce

**OBJECTIVES** – This study intends to determine the extent to which raw coal in pulverized form can be gasified within the environment of a freely propagating hydrogen-oxygen detonation. Should this mechanism be found viable, continuous high-rate high-capacity gasifiers using maintained

detonations in annular channels could be developed. These gasifiers would be much smaller than existing designs which would considerably reduce the capital cost of the primary gasifier and result in a somewhat reduced capital cost to the consumer. Thus, a single-shot detonation duct to approximate conditions in a continuous detonative gasifier is being designed and built to execute a series of feasibility experiments. An additional objective is to analyze the aerothermochemical interactions between coal particles and detonation products on a first-order level.

**RECENT WORK AND ACCOMPLISHMENTS** — A considerable amount of output has already been obtained from the analytical phase of this study. Relative gas/particle velocities and local thermodynamic conditions have been computed along coal-particle trajectories as functions of particle size, detonation expansion-wave length, and detonation stoichiometry. Significantly, this analysis has shown the persistence throughout the particle motion of very high relative velocities. The analysis has also been used as a basis for the design of the detonation duct. That design has now been substantially completed and the facility is under construction.

**PLANS FOR THE COMING YEAR** — The detonation duct and associated support equipment and instrumentation will be assembled and tested. Initial experiments with Pittsburgh seam bituminous coal particles in the size range of 20 to 200 micron diameter will be conducted using a hydrogen-rich detonation. Collected product samples will be analyzed to assess the degree of conversion. Samples of ash and partially converted coal particles will be collected and observed with a scanning electron microscope. The theoretical model will be improved. Particle ablation will also be studied.

#### **SUMMER INSTITUTE IN ENERGY ECONOMICS AND MINING ECOLOGY FOR EDUCATORS**

UNIVERSITY OF NORTH DAKOTA  
DOE - \$16,855  
5/1/77 - 12/31/77  
Principal Investigator - R. Kauffman

**OBJECTIVES** — This project provided basic training in energy economics for secondary level social studies teachers and in mining ecology for secondary level biology and natural science teachers that could be carried into energy-related public school curricula in North Dakota. The national importance of North Dakota's sizable lignite reserves has created considerable interest among the state's citizens in the social, economic, and environmental impacts of energy development. It is hoped that providing teachers with a basic understanding of the economics and ecology of energy development (especially coal) will lead to improved quantity and quality of instruction on energy problems.

**RECENT WORK AND ACCOMPLISHMENTS** — Twenty-three North Dakota teachers attended a

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## CHEMISTRY OF LIGNITE LIQUEFACTION

UNIVERSITY OF NORTH DAKOTA

DOE - \$551,400; UND - \$135,657

1/1/76 - 12/31/78

Principal Investigator - V. Stenberg

**OBJECTIVES** – This project seeks to compare samples of solvent refined coal (SRC) and solvent refined lignite (SRL), to aid in the development of analytical methods for SRC, SRL, and their heavy liquids, and to investigate the mechanism of solvent refining using carbon monoxide-water and carbon monoxide-hydrogen water blends of reducing gases. Catalysts for upgrading SRC and for carbon monoxide reductions are being studied.

**RECENT WORK AND ACCOMPLISHMENTS** – As determined by x-ray diffraction studies, SRL (Pittsburg & Midway) and SRC (Tacoma) have crystallites present in the solid phase with about 15 Å diameter with the SRL having an average stack of four aromatic planes separated by 3.7 Å. The SRC stack contains an average of three. Low-angle x-ray scattering studies on the SRL sample in pyridine solution show the molecules to have an average of 18 Å radius of gyration. SRL can be 90 percent converted by catalytic hydrogenation into 20 percent light liquids, 15 percent light oil, 20 percent heavy oil and 35 percent gases (Low, et al., 1976). A series of commercially available Ni-Mo, Co-Mo, Ni-W, Pt, and Cr catalysts on  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  supports and a series of synthesized Ni-Mo/fibrous  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  ( $\alpha$  or  $\beta$ ),  $\text{ZrO}_2$  and  $\text{SiO}_2$  catalysts have been tested in hydrotreating of SRL into liquids. The temperature (450°C) and pressure (3500 psi) have been optimized. Ni-Mo/ $\text{Al}_2\text{O}_3$  is the best catalyst overall, but it has been determined that on a surface area basis, the Ni-Mo/ $\text{ZrO}_2$  and Ni-Mo/ $\text{TiO}_2$  catalysts are extremely good. Ni-Mo/ $\text{ZrO}_2$  is particularly promising, probably as a result of the dual acid-base character of the  $\text{ZrO}_2$  support. Phenol, methyl, and ethyl benzenes, diarylethers, diphenylmethane and carbazole are difficult to reduce under  $\text{CO-H}_2\text{O}$  liquefaction conditions (3000 psi and 425°C). These conditions readily convert benzophenone, benzhydrol, bibenzyl, diphenylsulfide, phenylbenzyl ether, thioanisole, quinoline, and anthracene into products. For these model compound reductions, the presence of tetralin has little effect, and sodium carbonate has a positive effect on the benzophenone and quinoline reductions, little effect on the bibenzyl reaction, and a negative effect on the reductions of the two sulfur compounds. Related to  $\text{CO-H}_2\text{O}$  as a reducing medium, a host of metal oxides (possible in situ catalysts in coal) has been tested by ESR for activity in electron transfer processes to carbon monoxide to form surface bound CO-. Only the alkaline earth oxides and thorium oxide have shown activity ( $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{BaO}$ ,  $\text{SrO}$ ,  $\text{ThO}_2$ ). These oxides have one common feature—they all possess Lewis base sites (two electron donors) and reducing sites (one electron donor). It is believed that a mechanism for initial activation of CO is described by first rapid adsorption on Lewis base sites followed by migration to reducing sites.

**PLANS FOR THE COMING YEAR** – The gel permeation chromatographic separations will be continued for the characterization and structure determination of SRL and SRC. Carbon-13 NMR will be used as a characterization tool for SRC, SRL, and derived liquids. Chemical modification of SRL and SRC will be used to improve solubility and for functional group determination. Other model compounds for carbon monoxide reductions will be examined for products formed and efficiency of catalysts related to minerals present in lignite. Detailed surface chemistry of the carbon monoxide adsorbed radical on  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{SrO}$ ,  $\text{BaO}$ , and  $\text{ThO}_2$  using ESR as a tool will be studied.

## OXYGEN STOICHIOMETRY AND ANALYSIS OF COAL, LIGNITE, COKE, AND DERIVATIVES

NORTH DAKOTA STATE UNIVERSITY

DOE - \$49,135; NDSU - \$18,000

4/1/77 - 3/31/78

Principal Investigator - A. Volborth

**OBJECTIVES** – This project involves oxygen stoichiometry studies in coal, lignite, coke, liquefaction products, SRC coal, and the like. This work includes grinding, special weighing procedures, sample preparation for neutron activation at the University of California, heat treatment in vacuum and air, volatiles determination, thermogravimetry, and purchase and installation of a Cockroft-Walton Fast-Neutron generator of the KAMAN A-711 type to perform oxygen, silicon, nitrogen and other neutron activation research at North Dakota State University. An attempt is being made to improve and to re-evaluate the classical methods of coal analysis by providing complete material balances of solid fuels based on accurate oxygen determination and to provide new information to the coal scientist and engineer that would permit better controlled experiments in coal conversion and in fuel industry in general.

**RECENT WORK AND ACCOMPLISHMENTS** – The results of recent work have fully confirmed previous doubts about the adequacy of the ASTM-approved methods of coal analysis. It is now evident that better material balances, better ash analyses, and refined ability to double-check summations of ultimate coal analysis and to detect errors and deficiencies is achieved by adding oxygen to the list of the determined major constituents of coal. About 400 coal samples have been prepared, heat treated, and are being analyzed, and numerous material balances have been published with interpretation. New possibilities to evaluate quantitatively oxidation of coal during drying and storage, of separating the oxygen due to mineral matter and high-temperature and low-temperature ash, as well as correlation of viscosity of coal-derived liquids with oxygen content have been explored and are being evaluated.

**PLANS FOR THE COMING YEAR** – With the purchase and installation of the new neutron generator on this campus and the planned purchase and installation of the multichannel analyzer, the single-channel detector system, the sample transfer system, and other related instrumentation, all the analytical work can now be performed on this campus rather than spending the 6-months each year on the University of California campus, where an accelerator has been available for preliminary work during the last 2 years. The goals remain essentially the same as in original proposals and the schedules have remained nearly unchanged.

## STUDY OF COAL REACTIVITIES

CASE WESTERN RESERVE UNIVERSITY

DOE - \$301,368; CWRU - \$20,116

6/1/76 - 5/31/78

Principal Investigator - J.C. Angus, N.C. Gardner

**OBJECTIVES** – This project intends to measure coal and char reactivities in environments representative of commercial processes for coal gasification. The results will be used to provide a data base for modelling of large-scale gasification plants, for example, Synthane. Secondary objectives include determination of rate expressions and mechanistic interpretations for the coal gasification process at practical conditions and the determination of whether catalytic gasification can both increase reactivity and provide increased primary methane yields.

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**RECENT WORK AND ACCOMPLISHMENTS** — The second-generation thermobalance reactor was designed and constructed. The thermobalance employs a novel means for obtaining the kinetic data, in that the entire reactor plus contents will be weighed during the course of a run, rather than simply weighing a hanging basket within the reactor core. This procedure permits duplication of conditions existing within a commercial reactor much more closely than can be achieved with a conventional thermobalance. In particular, the product gas stream composition, the flow regime within the reactor, and the initial heatup period can be more representative of commercial-large scale reactors. The new reactor provides a unique method for measuring coal and char reactivities at practical operating conditions. The steam gasification of Synthane chars was performed in an existing integral reactor. Uncatalyzed chars and chars with alkali metal and alkaline earth catalysis were studied. In addition, extensive measurements of composition changes in the product gas sampling lines were performed.

**PLANS FOR THE COMING YEAR** — The second-generation thermobalance will be used to make accurate differential rate measurements for a series of chars at representative commercial operating conditions. These data will be used for construction of kinetic rate expressions using techniques developed in the laboratory. Particular attention will be paid to the influence of extent of carbon conversion and catalysts on activation energies, surface areas, and pore structure.

## ELECTROCHEMICAL DISPOSAL OF $H_2S$

CASE WESTERN RESERVE UNIVERSITY

DOE - \$38,893; CWRU - \$3637

8/31/77 - 8/30/79

Principal Investigators - C.B. Brosilow, J.C. Angus

**OBJECTIVES** — The purpose of this research is to obtain experimental data on the direct electrolysis of liquid  $H_2S$  to elemental S and  $H_2$ . The information on anodic and cathodic products, overvoltages, and current densities will be used to make a preliminary estimate of whether an electrochemical process for  $H_2S$  disposal, which will also deliver net electrical work, is technically and economically viable.

**RECENT WORK AND ACCOMPLISHMENTS** — Electrolysis of liquid  $H_2S$  at both cryogenic and room temperatures has been accomplished. Pyridine was used as an electrolyte. Gaseous hydrogen was evolved at the cathode and elemental crystalline  $\alpha$ -sulfur was the anodic product. The sulfur dissolves in the liquid  $H_2S$  which prevents the formation of an insulating layer of sulfur on the anode. Cathodic current efficiencies were  $99.8 \pm 0.7$  percent and anodic efficiencies were  $86.3 \pm 3.6$  percent. The cause of the low anodic efficiencies has not yet been identified. Parallel reactions or systematic errors in the analytical procedures are each possible. The major source of voltage loss is solution resistance.

**PLANS FOR THE COMING YEAR** — The principal goals in the coming year are to obtain a better measure of the current efficiencies and to investigate a selection of alternate electrolytes that might reduce the solution resistivities. Of particular interest are electrolytes that will be acidic (proton donors) to  $H_2S$ .

## RECOVERY OF ULTRAFINE COAL PARTICLES BY MICROFLOTATION

OHIO STATE UNIVERSITY  
DOE - \$20,000; OSU - \$8571  
8/1/77 - 9/30/78  
Principal Investigator - A.J. Rubin

**OBJECTIVES** — The purpose of the research is to examine the factors controlling the flotation of near-colloidal-size coal particles using extremely small bubbles at low gas rates. This approach, dispersed air microflotation, has been successfully applied to a number of colloidal particle types but not to coal. The generation of bubbles by dispersed air and dissolved air methods will be compared in a laboratory study. The advantage of dissolved air (pressure) flotation is the potential savings in the costs of chemicals needed to produce the small bubbles when using dispersed air microflotation. Flotation "domains," a graphical means of summarizing experimental data and aiding interpretation, will be constructed. The results will be related to adsorption studies and electrophoretic measurements.

**RECENT WORK AND ACCOMPLISHMENTS** — The initial studies are concerned with preparing a finely crushed oxidized coal and determining its colloidal properties in aqueous dispersions. Lump coal is coarse ground, balled milled for several hours, and sieved. The very fine fraction, less than approximately 300 mesh, is then oxidized in solutions of hydrogen peroxide. Unstable particles are settled out and the remainder is filtered and washed. These dispersions are being studied for their stability as a function of pH. Concurrently, a dispersed-air flotation apparatus is being constructed, and experimental techniques are being developed.

**PLANS FOR THE COMING YEAR** — Research on the flotation of the ultrafine coal will begin after completing the stability and electrophoretic mobility studies. Dispersed air flotation will be investigated initially. Parameters will include pH, collector and frother type and concentration, particle concentration, and gas flow rate. Colloidal-sized bubbles and coagulation, initially with aluminum sulfate, will be used to effect microflotation, a high-rate process. If time permits, these investigations will continue with dissolved-air (pressure) flotation. An attempt will also be made to obtain field samples of coal washings of various ash content for parallel studies.

## COAL HYDROGENATION VIA BONDING OF METALLIC COMPOUNDS TO COAL

UNIVERSITY OF CINCINNATI  
DOE - \$161,159; UC - \$10,580  
4/1/76 - 3/31/79  
Principal Investigator - M. Orchin

**OBJECTIVES** — This project intends to determine whether covalent bonding of a metal, such as tin, directly to the aromatic structures in coal will prove to be a better catalytic system for the hydrosolubilization of coal than the same quantities of Sn added in bulk to coal. If this proves to be the case, it will be a further objective to ascertain the reasons for the superiority.

**RECENT WORK AND ACCOMPLISHMENTS** — Cross-linked polystyrene beads have been used as a model for coal and the methods used for anchoring catalysts to such polystyrene have been successfully extended to coal. Results to date indicate that: (1) Coal undergoes electrophilic substitution reactions analogous to those of polystyrene; (2) tin (and other metals) can be covalently bonded to coal by using the experimental reactions; (3) tin covalently bonded to the coal, in the form coal -

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$\text{SnR}_3$ , is superior at the same concentration level to Sn added as  $\text{R}_3\text{SnCl}$ ; (4) the nature of R does not appear to influence results, i.e., when R is  $\text{CH}_3$ , it is as effective as R = phenyl; (5) covalent bonding of Sn directly to the aromatic structures in coal or through  $\text{CH}_2$ - groups is equally effective; and (6)  $\text{R}_3\text{SnCl}$ , although an effective catalyst for the hydrosolubilization of coal is ineffective in the hydrogenolysis of covalent bonds of pure compounds.

**PLANS FOR THE COMING YEAR** – Besides verifying the superiority of bonded metals as catalysts, efforts will be directed toward preparing synthetic “asphaltenes.” This work will involve modifying polystyrenes of known molecular weight by incorporating the number and kind of oxygen and nitrogen atoms known to be present in asphaltenes and determining the chemical and physical properties of such asphaltenes.

#### EROSION STUDY IN TURBOMACHINERY AFFECTED BY COAL AND ASH PARTICLES

UNIVERSITY OF CINCINNATI

DOE - \$164,218; UC - \$8825

8/1/76 - 7/31/79

Principal Investigators - W. Tabakoff, A. Hamed

**OBJECTIVES** – This program seeks to carry out experimental and theoretical studies of the erosion caused by coal and ash particles to potential turbine materials, to find the important parameters needed for basic understanding of the erosion phenomena, to study the aerodynamic effects on erosion of alloys for future turbines utilizing coal fuel, to develop a model for predicting particle trajectories, and to develop a computer program to facilitate the prediction of erosion in turbomachinery design.

**RECENT WORK AND ACCOMPLISHMENTS** – The design and construction of a high-temperature erosion wind tunnel were completed. Some erosion results from the alloys studied in the coal wind tunnel were obtained (aluminum, stainless steel, titanium).

**PLANS FOR THE COMING YEAR** – Experimental work in both erosion wind tunnels will continue to find important parameters that influence erosion caused by coal particles and ashes. Different coal ashes (from different parts of the United States) will be used to find the erosion damage in turbomachinery.

#### CATALYSTS FOR UPGRADING COAL-DERIVED LIQUIDS

OKLAHOMA STATE UNIVERSITY

DOE - \$318,842; OSU - \$22,893

6/9/75 - 3/8/78

Principal Investigator - B.L. Crynes

**OBJECTIVES** – This program investigates catalysts for upgrading liquids from coal-to-oil processes to remove sulfur, nitrogen, and possibly other heteroatoms. Catalysts with supports of various pore sizes and pore size distributions will be tested to seek optimum pore properties for such upgrading processes. Based on these studies, catalysts will be recommended for upgrading coal-derived liquids under hydrogen pressure to remove certain heteroatoms.

**RECENT WORK AND ACCOMPLISHMENTS** – Continued catalyst assessments (~15, both commercial and laboratory prepared) have been made in two trickle-flow reactors to study the effects of



catalyst support properties on sulfur and nitrogen removal from coal-derived liquids. Emphasis has been on determining effects of changing the support pore size, pore distribution, and total surface of cobalt-molybdenum-on-alumina catalysts. Six coal liquids have been processed, raw anthracene oil used as a reference feed, an FMC oil from Project COED, an oil from an SRC process, Synthoil liquids, a hydrocarbonization liquid, and a mixture of a Synthoil plus anthracene oil. An automated catalyst life-test unit consisting of a bank of three trickle-flow reactors has been used to assess and study the activity decay rate and mechanisms of catalysts as they hydroprocess coal liquids. Continuous experimental runs of up to 700 hours of oil-on-catalysts have been made. The designed experimental set on one catalyst and a special coal liquid has been completed, and the product oils plus the catalysts are now being assessed in detail using mass spectroscopy, ESCA, AUGER, and other sophisticated techniques. The work on characterization of coal liquids has continued in identifying those compound species and types that affect the overall hydroprocessing sequence. Also, additional analytical instrumentation and methods have been developed.

**PLANS FOR THE COMING YEAR** — The project will terminate during March 1978. No further reactor experimental runs will be made; however, the detailed analytical assessment and data interpretation are scheduled during these last few months.

#### INTERACTION OF H-ATOMS WITH ULTRAFINE COAL DUST

OKLAHOMA STATE UNIVERSITY

DOE - \$45,000; OSU - \$3735

5/16/77 - 5/15/78

Principal Investigator - G.J. Mains

**OBJECTIVES** — This project seeks to identify and quantify the gasoline-type hydrocarbons produced by the interaction of H-atoms with very fine coal dust above 175°C. The quantitative effects of temperature, H-atom concentration, extent of conversion, coal particle size, and concentration on product yields are the primary objectives. Deductions regarding the surface and bulk molecular structures of various coals based on the chemical composition of the observed products are secondary objectives. Both the primary and secondary objectives have obvious relevance in the design of third-generation coal gasification and liquification processes as well as in providing guidance for feedstock selection for ongoing and second-generation plants.

**RECENT WORK AND ACCOMPLISHMENTS** — Equipment has been assembled for bench-scale crushing and size classification of the coal dust. It was found that crushing and sieving in an inert atmosphere was necessary to prevent surface oxidation and size classification down to 5  $\mu\text{m}$  in a nitrogen atmosphere is now feasible in gram quantities. The photoreactor was modified to permit operation to 300°C at low flow rates of  $\text{H}_2$ , but temperatures in the range of 175° to 200°C are

identifying the primary intermediates cracked off coal by very rapid H-atom T.O.F. mass spectrometry was abolished when the instrument broke down and required extensive electronic updating. Progress toward the secondary objective of coal structure elucidation is being made via an ancillary flash-heating extractive solvent quenching program.

## PHASE EQUILIBRIUM AND VOLUMETRIC PROPERTIES OF COAL-DERIVED FLUIDS

OKLAHOMA STATE UNIVERSITY

DOE - \$199,640; OSU - \$10,500

3/15/76 - 3/14/79

Principal Investigator - R.L. Robinson, Jr.

**OBJECTIVES** – The thermodynamic properties of fluid mixtures are required for accurate design of equipment in which such fluids are stored, transported, or separated. Selected property data are being obtained for components and conditions where information is lacking. The mixtures studied will approximate those which occur in the processing of coal-derived fluids. Research is divided into three major areas: (1) Design, construction, and operation of a Burnett-type apparatus for measurement of volumetric properties (pressure-volume-temperature-composition relationship) of gases. Measurement of the properties of the pure substances and selected mixtures of  $H_2$ , CO,  $CO_2$ ,  $CH_4$ , and  $H_2O$  at conditions from ambient to 800°F and 2000 psia (or to the maximum temperature at which chemical reactions are unimportant). (2) Design, construction, and operation of a variable-volume, windowed phase-equilibrium cell capable of operation to 800°F and 2000 psia. Measurement of vapor-liquid equilibrium in mixtures composed of selected light hydrocarbons/heavy aromatic hydrocarbons/water. (3) Testing and/or development of prediction methods for estimation of volumetric properties and phase equilibrium in mixtures of the type studied experimentally. Presentation of results in formats suitable for use in process design calculations.

**RECENT WORK AND ACCOMPLISHMENTS** – The two separate experimental facilities have been designed and constructed. The Burnett-type volumetric property apparatus is completely constructed and under testing. Temperature stability to  $\pm 0.03^\circ F$  at temperatures to 400°F has been established in the gas bath in which the Burnett equipment is housed. Tests are underway to establish the precision of the pressure-volume-temperature-composition measurements that can be made with this facility. The variable-volume, windowed phase equilibrium apparatus has been fabricated; the high-temperature, rocking gas bath in which the cell will be housed is in the final stages of construction.

**PLANS FOR THE COMING YEAR** – Data will be obtained using the Burnett apparatus on the volumetric properties of the pure substances  $H_2$ , CO,  $CO_2$ ,  $CH_4$ , and  $H_2O$  and the 10 binary mixtures they form. Later, data will be obtained on selected mixtures containing three to five of these components that will be used to test and develop models for representing the volumetric properties of these systems and the thermodynamic properties derived therefrom. Construction of the phase equilibrium facility will be completed, and the apparatus will be tested by measurement of phase equilibrium for a suitable binary system. Data will then be taken on a multicomponent system containing several components (e.g., methane, ethane, butane, toluene, naphthalene) selected to model multicomponent coal-derived fluid systems.

## CHARACTERIZATION OF COAL-DERIVED LIQUIDS AND OTHER FOSSIL FUEL MATERIALS EMPLOYING MASS SPECTROMETRY

OKLAHOMA STATE UNIVERSITY

DOE - \$289,206; OSU - \$107,674

9/30/76 - 9/19/79

Principal Investigator - S.E. Scheppele

**OBJECTIVES** – This project intends to develop new and/or to refine mass spectrometric techniques for obtaining both routine and detailed characterization data for coal-derived liquids and other fossil-fuel-related materials. Characterization data are imperative for both developing and routinely monitoring commercial coal liquefaction and gasification processes as well as for the upgrading and refining of such fluids. Mass spectrometric characterization research will contribute to areas such as: development of superior solvents in coal-liquefaction processes; catalyst development, deactivation, and regeneration; development of correlations to predict thermodynamic properties; determination of factors controlling the stability of coal-derived liquids, choice of materials for reactor fabrication; on-stream process monitoring; identification and quantification of substances of environmental and biological concern; production and refining of products obtained from other fossil-energy sources such as heavy ends of petroleum, heavy oils, shale, and tar sands; and the mapping of known and the location of new fossil-energy reserves.

**RECENT WORK AND ACCOMPLISHMENTS** – Fourteen industrial, governmental, and university laboratories involved in fossil-energy research were visited to assist in acquiring information for preparing a report on mass spectrometry and fossil-energy conversion technology. Existing mass spectrometer facilities were augmented as follows. A commercial data acquisition and processing system was configured and interfaced to both the CEC 21-110B double-focusing mass spectrometer and the OSU IBM 370/158 computer. A comparator/microdensitometer was made manually operational. A digital integrator, an encapsulated sample injector, and a capillary sample splitting injector constitute acquired ancillary gas-chromatographic equipment. The combination field-ionization/electron-impact ion source was modified to operate in the field-desorption mode. Construction of a temperature control-display module for the direct-introduction probe inlet system is essentially complete. Field-ionization mass spectrometry, FI/MS, which produces virtually fragment-ion-free mass spectra, was previously shown in the laboratory to be ideally suited to quantitative analysis of aromatic components of coal-derived liquids. Extension of the technique to the analysis of saturates would be significant because FI/MS would in contrast to electron-impact MS provide a molecular-ion-carbon-number group-type quantitative analysis of these fractions. The previously reported dependence of the relative sensitivities for FI of saturated hydrocarbons in the presence of low-molecular weight aromatic hydrocarbons on mixture composition suggested that such an extension might not be feasible; however, we demonstrated that as the FI emitter temperature is increased from 100 to 270°C, the FI sensitivity for heptane relative to benzene decreases for any given binary mixture and exhibits a decreasing dependence on binary mixture composition. The relative sensitivity was found to be composition independent at an emitter temperature of 270°C. Similar results were then shown to attain for a number of multicomponent mixtures containing a variety of saturated and aromatic hydrocarbons. Composition-independent FI sensitivities were also determined at 270°C emitter temperature for 20 alkanes and naphthenes in 22 mixtures containing only saturated hydrocarbons. Based upon these results, it seems reasonable to conclude that FI/MS can be developed as a rapid and routine technique for obtaining molecular-ion group-type analysis of saturate fractions and that medium-resolution FI/MS constitutes an

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approach to the quantitative analysis of mixtures containing both saturates and aromatic neutrals without the need for separating them prior to analysis.

**PLANS FOR THE COMING YEAR** – Preparation of the report on MS technology will continue. Performance of the data acquisition system-mass spectrometer combination will be optimized. Establishment of interactive communications between the minicomputer and the IBM 370/158 reflects resolution of RJE 80 software problems by the minicomputer manufacturer. Work will proceed with interfacing the comparator/microdensitometer, direct-probe-control module, and gas chromatographic equipment to the minicomputer. Software modification and development at both the Fortran and assembly language levels will provide for computer acquisition, reduction, and assisted interpretation of data from both the mass spectrometer and peripheral instrumentation. Development research will also emphasize: FI/MS for quantitative analysis; FI/MS in combination with micromolecular probe distillation for quantitative analysis; field-desorption MS for qualitative analysis of relatively nonvolatile high molecular weight compounds; simulated distillation in combination with FI/MS.

#### WORKSHOP ON ENERGY RESOURCES

OREGON STATE UNIVERSITY  
DOE - \$11,725; OSU - \$6893  
1/1/76 - 12/31/76  
Principal Investigator - C.H. Wang

**OBJECTIVES** – The 1976 Summer Workshop on Energy Resources and Electric Power Generation sought to transmit to high school and junior college science teachers reliable information on: energy consumption patterns; the importance of energy conservation; availability of alternative energy resources; status of coal technology and its environmental impact; status of nuclear energy development and its environmental impact; and a comparative evaluation of various energy alternatives.

**RECENT WORK AND ACCOMPLISHMENTS** – Forty participants selected from 106 applicants (high school and junior college science teachers) participated in the 5-day workshop. Seven outside speakers and six OSU speakers presented 13 papers. Participants were enthusiastically involved in discussion and felt that work of this type is of great importance in providing reliable information to high school and junior college science teachers.

**PLANS FOR THE COMING YEAR** – Work under this contract has been concluded.

#### HEAT TRANSFER IN HIGH-TEMPERATURE FLUIDIZED BEDS WITH IMMERSED TUBES FOR COAL GASIFICATION SERVICE

OREGON STATE UNIVERSITY  
DOE - \$149,128; OSU - \$17,597  
9/30/77 - 9/30/80  
Principal Investigator – J.R. Welty

**OBJECTIVES** – This research deals with heat transfer between a high-temperature ( $\sim 1100^{\circ}\text{K}$ ) gas fluidized bed and immersed tubes. The first objective is to develop an analytical model that will predict the local heat transfer rates and tube-surface temperatures as functions of bed operating parameters. The second objective is to perform experiments with an operational high-temperature bed to support the analytical model and to provide data on high-temperature operation.

**RECENT WORK AND ACCOMPLISHMENTS** – Because of the short time the contract was in effect during the reporting period, the work accomplished was the initiation of the analysis and the refinement of apparatus design.

**PLANS FOR THE COMING YEAR** – Analytical model development will proceed. Radiant heat exchange between the hot particles and the cylinder is to be evaluated using the net radiation method assuming that the particle strings adjacent to the cylinder wall and at the bubble boundary form isothermal gray surfaces. Bubbles are to be treated by solving Laplace's equation for pressure using the method of images and representing a bubble as a doublet. The pressure solution will be used to obtain average gas velocity within bubbles utilizing mass conservation. The analytical approaches described will be used to develop computer programs for local and mean heat transfer coefficients. Design of the fluidized-bed test facility and related components will continue. The major-system component designs are complete except for the combustion control system for hot gas supply, the electronic circuitry for combustion startup and operational sequencing, and the control panel console. Construction of the experimental facility will begin as soon as final designs are completed and will be finished during FY 1978. No delays should be encountered for lack of components.

## RECIRCULATING BED REACTORS FOR COAL PROCESSING

CARNEGIE-MELLON UNIVERSITY  
DOE - \$677,075; CMU - \$35,636  
7/1/76 - Continuing  
Principal Investigators - T.W. Bierl, M.J. Massey

**OBJECTIVES** – This program intends to facilitate the application of recirculating bed reactors to the processing of coal. Two aspects of recirculating bed reactor (RBR) technology are addressed: one equipment oriented and the other process related. An RBR is a device that continually circulates a portion of its solid inventory around a vertical loop. The driving force solids circulation is the static head of solids in the reactor standleg, and the energy for solid transport is provided by the process fluid that contacts the solid inventory for reaction purposes. Task 1 "Coal Mixer Modelling Studies" will initially involve the experimental investigation of the mass and heat transfer problems associated with mixing streams containing entrained solids. A 3-inch-diameter plexiglas model of an RBR is being used for these studies. Task 2 "Coal Hydrodesulfurization Studies" has a process objective and has only limited RBR hardware considerations. It is the goal of these studies to demonstrate significant organic sulfur reduction in coal by thermal processing in hydrogen and mapping the effect with variations in pressure, temperature, reactor residence time, and feed gas composition in a well-characterized experimental reactor. An experimental reactor called the HDS (hydrodesulfurization) reactor has been constructed for this purpose.

**RECENT WORK AND ACCOMPLISHMENTS** – The design, construction, and debugging of the RBR cold model for coal mixer modelling studies has been completed. Several instrument development programs were required to perform the mixing experiments: the wall shear stress analyzer, the sampling/momentum probe, feed solids tagging procedure, and solids sample analysis. These systems have now been brought onstream, and their performance characterized. Implementation of the mixing experiments plan is now in progress. The initial experiments will study isokinetic injection in a concentric geometry which will provide data on the self-mixing characteristics of the fast bed. This base case will then be perturbed to study the effects of those conditions theorized to be

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favorable to mixing enhancement. Design, construction, and debugging of the HDS Reactor for Coal Hydrodesulfurization Studies, has been completed, and only minor adjustments are necessary before trial tests are taken.

**PLANS FOR THE COMING YEAR** – Task 1 will be dedicated to quantifying the characteristics of RBR coal mixers. The experiments are expected to characterize dispersion of gas, solid, and heat occurring during mixing processes. Experiments determine only the degree of solids mixing that occurs. Momentum and solids flux profiles will also be obtained. Task 2 will be devoted to completing shakedown of the experimental apparatus and operation of the unit to gather experimental data on coal sulfur chemistry.

## CATALYTIC SYNTHESIS OF GASEOUS HYDROCARBONS

CARNEGIE-MELLON UNIVERSITY

DOE - \$206,481; CMU - \$7592

5/75 - 8/78

Principal Investigator - A.L. Dent

**OBJECTIVES** – This work was undertaken with the objective of developing potential Fischer-Tropsch catalysts with higher selectivity for synthetic petrochemical feed-stocks, preferably the C<sub>2</sub>-C<sub>4</sub> olefins. Since this process is inherently similar to methanation, it is also anticipated that during the implementation of this work, a greater understanding of the factor affecting the selective catalytic hydrogenation of carbon monoxide in general will evolve.

**RECENT WORK AND ACCOMPLISHMENTS** – Several iron- and cobalt-based Fischer-Tropsch catalysts have been investigated. In the iron-copper series (100 Fe:X Cu:100 kieselguhr: Y K<sub>2</sub>O), the content of copper and alkali were systematically varied. The results indicated that the product distribution shifted towards lower molecular-weight hydrocarbons as the copper content, the olefin yields, and the alkali content increased. In the more extensive tests of the cobalt series which were conducted in the Bertly reactor unit (CRU-2), the effect of constant amounts of Group IV to VII metals was studied. These results showed that cobalt-manganese-alumina catalysts promoted with alkali yielded a C<sub>2</sub>-C<sub>4</sub> (essentially ethylene and propylene) olefin absolute selectivity greater than 25 percent; however, on a methane-free basis, their selectivities are greater than 50 percent. In general, these results indicated that the product distribution from these cobalt catalysts is critically dependent on the alkali content, while overall activity depends on the level of metal loading. Thus, there is considerable encouragement in efforts to fulfill the objective of selective production of C<sub>2</sub>-C<sub>4</sub> olefins.

**PLANS FOR THE COMING YEAR** – The studies of cobalt-manganese FT catalysts will be continued with the inclusion of additional tests at higher conversion levels, longer on-stream test periods, the different H<sub>2</sub>/CO feed ratios. In addition, detailed kinetic and mechanistic studies of these catalysts will be undertaken, using infrared spectroscopic techniques applied to catalytic systems. Other studies involving nickel aerosol catalyst characterization will be completed.

## COAL AND COAL REFUSE FOR MANUFACTURE OF NATURAL CEMENTS

CARNEGIE-MELLON UNIVERSITY

DOE - \$19,900

8/1/77 - 7/31/78

Principal Investigator - G.M. Krokosky

**OBJECTIVES** – This research seeks to use the large supply of waste coal that is being produced in the Western Pennsylvania region for the production of cement. This waste-coal primarily consists of shale and low-Btu coal. The heating content of the material is around 2000 to 3000 Btu/lb. The remainder of the material consists primarily of alumina and silica. There is no feasible use for the material, and it sits in “gob piles” in Western Pennsylvania. Occasionally, these piles catch on fire and the resulting material is known as red-dog. The objective of the research is to use this material as a feed stock for the production of low-cost Portland cement. The alumina and silica content of this material is high enough to be used either directly with limestone or some calcium-bearing rock. The material would be used in the vertical-kiln production-process for Portland cement. This process is extremely efficient in the utilization of energy.

**RECENT WORK AND ACCOMPLISHMENTS** – Work has involved analyzing the constituents of the Western Pennsylvania coal and the constituents of various feedstock materials that would be readily available in this and other areas. A vertical kiln is being constructed to try out the process. The process, which has been demonstrated experimentally in Colorado on a small kiln, proved to be very successful, although there are some problems involved in extracting the material from the kiln. These problems are basically physical and can be worked out. Negotiations are underway with the Ideal Cement Company in Fort Collins, Colorado to acquire their vertical kiln. Moving the kiln will be a major problem although costs of moving will more than offset the cost and time for reconstructing a new kiln.

**PLANS FOR THE COMING YEAR** – Because of certain time delays in shipping the kiln from Fort Collins, it might be necessary to construct a smaller version of this vertical kiln at Carnegie-Mellon University. In the meantime, small-scale testing is being carried out involving mixtures of cement, rock, and coal stock material from various sources. Either straight limestone rock or lime material can be used as a feed rather than cement rock. The advantage of natural cement rock is its low silica content, which reduces the temperature for calcination. Full pilot-scale operations will begin at the end of March 1978, or earlier if the smaller C-MU built kiln is obtained.

## MECHANISMS OF THE HYDROGEN SULFIDE-DOLOMITE REACTION

CARNEGIE-MELLON UNIVERSITY

DOE - \$231,259; CMU - \$12,171

7/1/76 - 6/30/79

Principal Investigator - K. Li

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**RECENT WORK AND ACCOMPLISHMENTS** – A high-pressure thermogravimetric analysis system including a DuPont 951 Thermogravimetric Analyzer and a DuPont 990 Thermal Analyzer has been designed and constructed. The system is designed for operations up to 1000 psia and 1000°C. A separate gas feed system provides for the feeding, blending, and metering of up to six different gases. Kinetic data have been obtained for the half-calcination of Gibsonburg, Ohio dolomite in the completed TGA system at temperatures of 700° to 800°C for each of the pressures: 1.0, 6.1, 10.2, 20.4, and 27.2 atm. In addition, data for a Canaan, Connecticut dolomite were obtained at 20.4 atm. Pore and grain structures of reacted samples were studied by nitrogen adsorption, mercury porosimetry, and scanning electron microscopy. The distribution of the reaction product determined for several partially reacted pellets of the Ohio dolomite by optical microscopy indicates that the reaction occurs topochemically at the grain level. The conversion-time data appear to follow a relationship similar to that for a solid-solid transformation with nucleation of the product phases as the rate-controlling step. This mechanism is not inconsistent with the observed morphological behavior. Experiments have been conducted in the TGA system for the sulfidation of the Ohio dolomite in 7.8 atm of 0.8 percent H<sub>2</sub>S in N<sub>2</sub> and in 14.6 atm of 1.0 percent H<sub>2</sub>S in N<sub>2</sub>. Pore and grain structures as well as compositional profiles have been determined for selected samples. Although the reaction is not completely topochemical on either the pellet or the grain level, as indicated by the structural data, an unreacted-core model with a first-order reaction step being rate-controlling fits the conversion-time data reasonably well.

**PLANS FOR THE COMING YEAR** – Measurements of reaction rates in the TGA system for sulfidation and carbonation together with determinations of structural and compositional profiles of reacted samples will continue. In addition to the Ohio and Connecticut dolomites, two other dolomites of different geographical origins will be tested. Kinetic models based on rate and structural data for half-calcination and sulfidation will be further developed as more data become available. Mathematical modeling of the carbonation reaction will be initiated in parallel with data collection.

#### ADVANCED METHANOL SYNTHESIS CATALYSTS

LEHIGH UNIVERSITY  
NSF and DOE - \$151,200  
3/1/75 - 2/28/77  
Principal Investigator - K. Klier

**OBJECTIVES** – This program seeks to investigate advanced catalytic systems for the synthesis of methanol and methyl fuel from coal-generated syngas. Main emphasis is on the low-temperature and low-pressure performance of the catalysts. Further research will be devoted to the improvement of sulfur tolerance of the investigated catalytic systems. The benefits of the new methanol synthesis would be to produce economically priced, pollutionfree fuel for automotive transportation, power generation, and a basic chemical for plastic manufacture utilizing U.S. coal reserve as a raw material.

**RECENT WORK AND ACCOMPLISHMENTS** – The results of this work are: catalysts of lower surface area and more stable than commercial catalysts but equally active and selective were synthesized; the Cu/ZnO/alumina or chromia catalysts and their precursors were fully characterized by diffraction and electron spectroscopic methods, and aluminum and chromium were found to enhance the Cu<sup>I</sup> concentration in Cu<sup>I</sup>/ZnO; the catalytically active component of the low-pressure Cu/ZnO/alumina or chromia catalyst has been identified to be the Cu<sup>I</sup> solution in ZnO (1:10); the



effect of oxygen in the feed gas was found to increase methanol yields through oxidizing copper metal and increasing the level of  $\text{Cu}^{\text{I}}$  in the active phase  $\text{Cu}^{\text{I}}/\text{ZnO}$ ; there is an optimum concentration of  $\text{CO}_2$  in the feed gas for each catalyst composition, the effect being, as in previous cases, to optimize the  $\text{Cu}^{\text{I}}/\text{ZnO}$  composition; and the effects of sulfur and chlorine containing gases are to poison the  $\text{Cu}^{\text{I}}$  centers with the exception of COS whose effect is similar to that of  $\text{CO}_2$ , i.e., to stabilize the synthesis, rather than to that of  $\text{H}_2\text{S}$ . The low-surface area catalysts prepared herein are suitable for liquid phase methanol synthesis, which is economically superior to the gas phase synthesis. The stabilization of the synthesis by COS and improvement by oxidizing gases provide a promising route for circumventing the sulfur sensitivity of the catalyst.

**PLANS FOR THE COMING YEAR** — A new project, "Methanol and Methyl Fuel Catalysts," has been proposed. The findings that oxidizing gases and COS stabilize the synthesis and the active phase in  $\text{Cu}^{\text{I}}/\text{ZnO}$  system were instrumental in proposing studies leading to optimization of the feed gas composition, catalyst supports, and further promoters. In addition, studies of catalysts promoting the synthesis to methyl fuel rather than to pure methanol were proposed.

A further objective of this research is to provide, on the basis of research results, ratings, and recommendations of methanol and methyl fuel catalysts for diverse processes such as liquid-phase methanol synthesis, coal gasification-methanol integrated processes, and coal gasification-methanol-gasoline integrated process.

## CENTRIFUGAL FLUIDIZED COMBUSTION OF COAL

LEHIGH UNIVERSITY

DOE - \$138,997

9/17/76 - 9/16/78

Principal Investigators - E.K. Levy, J.C. Chen

**OBJECTIVES** — Conventional fluidized-bed combustors, with the bed material fluidized against the force of gravity, have many desirable features; however, for large-capacity power-generation applications or with very fine bed material, these systems require extremely large distributor areas, causing difficulties with startup, solids feed, and bed mixing. The centrifugal fluidized bed rotates about its axis of symmetry, and the fluidizing air flows radially inward through the porous cylindrical surface of the distributor. The inward drag force of the fluidizing air on the bed material is balanced by the large radial accelerations caused by the rotational motion, permitting much larger air flow rates per unit volume than are possible with a conventional fluidized bed operating against gravity. By varying the speed of rotation of the bed, the flow rate of air, and the bed temperature, it should be possible to achieve considerable variation in system power output providing the capability for operating over a wide range of part load conditions. In addition, the added flexibility due to bed rotation and the small size of the system should ease the problem of startup. With a bed material of dolomite or limestone to capture  $\text{SO}_2$ , the centrifugal combustor could be used to burn high-sulfur coal or coal char.

The centrifugal combustor would be operated as an adiabatic device with sufficient excess air to maintain bed temperatures in the desired range. For power generation applications, it might be used as the combustor in a combined gas turbine/steam turbine cycle.

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The successful development of the centrifugal fluidized bed concept would provide a system for coal or char combustion which would be compact, clean, efficient and would have the capabilities of being operated at full or part load conditions. The system might be used for utility size plants or for smaller industrial power generation applications.

The objective of the program is to determine the feasibility of operating a centrifugal fluidized bed in a continuous mode. The operating constraints as affected by limitations in fluidization, elutriation, and solids handling are being determined.

**RECENT WORK AND ACCOMPLISHMENTS** – The investigation consists of a series of experiments using a model of a fluidized bed combustor operated at room temperature and pressure. Fluidized bed materials that simulate coal and coal char in specific gravity, particle size, and distribution are being used to determine the requirements for minimum fluidization, bed pressure drop, freeboard pressure drop, and the extent of particle elutriation. The constraints affecting the addition and removal of material from the bed are being determined.

A second set of experiments is being performed to study the fluid mechanics of confined vortex flows without particles. This work is intended to develop criteria for the flow regimes in which significant secondary flow patterns occur. Once the proper criteria are established, comparisons will be made with the experiments on rotating fluidized beds to determine if the nonuniformities affect the bed stability and the quality and uniformity of fluidization.

During the past 15 months, a 12-in.-diameter by 6-in.-high model was fabricated, and experiments on bed startup and fluidization were performed using glass beads in the 121 to 475  $\mu\text{m}$  range. Experiments with cylindrical and conical distributors indicate it is difficult to start the bed from a slumped stationary position unless the distributor is tapered. With a slightly tapered distributor, the bed material readily distributes itself over the entire height of the test section and achieves a fluidized state. The relationship between bed pressure drop and air flow is qualitatively similar to that of a conventional fluidized bed with apparent values for  $\epsilon_{mf}$  in the range of 0.38 to 0.45. The experiments indicate strong effects of distributor angle, grid pressure drop, angular velocity, particle size, and bed mass on startup, minimum fluidization, and bed pressure drop. Theoretical analyses, developed to predict the influence of these factors on system performance, are generally in good agreement with experimental observations.

Two additional test sections were designed and built to study solids feeding and removal. Initial results suggest it is possible to feed solids continuously to a rotating bed and remove solids in a continuous manner.

The study of the flow patterns and velocity profiles of the gas flow within the centrifugal fluidized bed was initiated. The test section and velocity probe traversing mechanism were built; the hot film fiber anemometer probes were calibrated; and development work began on a computer procedure for data reduction and analysis.

**PLANS FOR THE COMING YEAR** – Additional batch experiments are planned to gather more data on fluidization and particle elutriation. The work on solids feeding and removal will be continued to develop quantitative relationships between feed and removal rates and the system

design and operating parameters. The calibration, data reduction, and analysis program for the anemometer probes will be completed and detailed studies of the fluid mechanics of the confined vortex flow system will be made.

## FRACTURE MECHANICS AND SURFACE CHEMISTRY STUDIES OF STEELS

LEHIGH UNIVERSITY  
DOE - \$294,920; LU - \$58,532  
9/17/76 - 9/16/79

Principal Investigators - R.P. Wei, G.W. Simmons

**OBJECTIVES** – Steels used in coal gasification vessels and piping are exposed to mixtures of hydrogen, water vapor (steam), hydrogen sulfide, methane, carbon monoxide, carbon dioxide, and other gases over a wide range of temperatures and pressures. Such mixtures, under certain operating conditions, can either enhance or inhibit subcritical crack growth in these steels that could result from exposure to hydrogen and hydrogenous gases in these mixtures. Data are needed to aid in the establishment of design and operational guidelines for assuring safe and reliable operation of coal gasification systems. This program will determine the correlation between the thermodynamics and kinetics of surface film formation and reduction in various gas mixtures and the kinetics of crack growth in these same mixtures.

**RECENT WORK AND ACCOMPLISHMENTS** – Principal efforts were devoted to material acquisition and qualification, equipment procurement and development, calibration of experimental techniques, and preliminary experimentation. A 2-1/4Cr-1Mo (ASTM A542, Class 2) steel has been selected for study. A 1-in.-thick plate of 2-1/4Cr-1Mo steel made from electric furnace heat has been procured from Lukens Steel Company and the chemical composition determined. The plate was heat treated and impact properties measured; they conform to ASTM A542, Class 2 specifications.

An Auger electron spectrometer (AES) with attachment for x-ray photoelectron spectroscopy (XPS) was installed in March 1977 and is fully operational. A special reaction chamber, suitable for operation at pressures up to 1000 psig and specimen temperatures up to 1000°F, has been designed and is being assembled. This reaction chamber incorporates suitable vacuum interlocks and specimen transport mechanisms for use with the AES/XPS system and will allow for the exposures of specimens to appropriate atmospheres and test conditions and their subsequent examination by AES and XPS. A special environment chamber and associated pressure and environment monitoring instrumentation, suitable for operation at pressures ranging from UHV to about 25 psig, has been procured for use in the fracture mechanics experiments. This chamber has been delivered and is now in operation. Initial fatigue crack growth experiments have been carried out in air and in dehumidified hydrogen over a range of  $\Delta K$  values from 20 to 60 ksi $\sqrt{\text{in}}$  (22 to 66 MPa $\sqrt{\text{m}}$ ). Data obtained in air (having a relative humidity of 40 to 60 percent at room temperature) show a small effect of temperature on fatigue crack growth. Crack growth rates increased with increasing temperature; the rates at 475°K (395°F) being approximately 50 percent faster than those at 295°K (72°F). Hydrogen (at about 5 psig) increased the rate of fatigue crack growth; the extent of this increase depended on stress intensity level, loading frequency, and temperature. Greater enhancement was observed at room temperature than at 400°K (260°F), and at 5 Hz as compared to 10 Hz. To provide background information for the surface chemistry studies, experimental calibration of the sputtering rate of oxide from surfaces of 2-1/4Cr-1Mo steel has been made as a function of ion beam energy and ion current density.

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**PLANS FOR THE COMING YEAR** — The experimental phases of the program will be continued. Experimental work will: determine the surface reaction kinetics and the susceptibility of 2-1/4Cr-1Mo steel to fatigue crack growth in pure, single-component gases ( $H_2$ ,  $H_2O$ ,  $H_2S$ , and  $CH_4$ ) from room temperature to about 700°K to establish baseline data for assessing surface reaction and crack growth responses in gas mixtures; establish and confirm conditions (primarily ratios of partial pressures and temperature) for surface film formation and reduction in  $H_2O/H_2$  and  $H_2S/H_2$  mixtures, as predicted by available thermodynamic data; determine if the susceptibility to crack growth and the kinetics for crack growth in hydrogen that is present in these mixtures are altered by changing conditions from that for surface film formation to that for film reduction as defined by the aforementioned surface chemistry experiments. Information from the experimental program will be used to develop guidelines for the design and operation of coal gasification systems and for correlation with other programs in furthering the understanding of hydrogen embrittlement mechanisms.

## MINERAL MATTER DISTRIBUTION IN PULVERIZED FUEL COALS

PENNSYLVANIA STATE UNIVERSITY

DOE - \$192,239; PSU - \$10,129

4/1/76 - 3/31/79

Principal Investigators - L.G. Austin, R.H. Essenhigh

**OBJECTIVES** — The slagging of mineral matter in coal when it is fired into a boiler furnace is being studied to develop tests to show when a particular coal is likely to cause slag deposit buildup near the furnace exit. When coal is burned as a pulverized fuel in power plant boilers, minimum cost and high efficiency are obtained by a high rate of heat release in the boiler furnace, along with high steam pressures and temperatures. However, the high temperatures cause adherence and accumulation of large masses of slagged ash in the upper part of the furnace that can break off and damage the bottom tubes of the furnace.

**RECENT WORK AND ACCOMPLISHMENTS** — The plane flame furnace burning about 20 lb/hr of pulverized coal has been redesigned to reduce heat losses and is being recalibrated. By controlled adjustment of the coal rate it is possible to obtain a range of heat release rates, and the furnace can switch from a nonslagging to a fluid-slag condition. Probes have been designed to collect adherent ash deposits on steel substrates at temperatures ranging from chilled to 700°C. By melting in a simple electric furnace it has been demonstrated that coal ash slags will not wet carbon, but the contact angle is decreased for slag compositions with high iron content. A new electric furnace is being constructed for controlled tests of the melting, wetting, and adherence behavior of coal ashes, synthetic mixtures of minerals, and coal ash enriched in certain components such as pyrite. The design allows a slag droplet to be produced at a desired melting temperature and lowered to touch a cooled metal substrate to see if it will wet and adhere. The behavior is viewed with an optical microscope system. Preliminary experiments with slag droplets formed in an oxyacetylene flame show that it is difficult to get coal ashes to adhere to stainless-steel substrates until the steel temperatures become close to the melting point of the slag. Progress has been made toward developing a computerized SEM technique for the analysis by the Colby program of Fe, S, Ca, K, Si, Al, and Ti in pulverized coal particles. It has been demonstrated that the mineral matter composition varies widely from one particle to another.

**PLANS FOR THE COMING YEAR** — When the furnaces have been constructed, the melting and adherence properties of a range of coal ashes will be studied as a function of metal substrate

temperature and chemical composition of the slag, including synthetic additions to the coal ash. The results will be correlated with the standard slagging behavior in the combustor. The SEM technique will be developed further to provide a guide to the variety of sizes and compositions of slag droplets expected from a given coal.

## **FRACTURE OF REFRACTORY CONCRETES**

PENNSYLVANIA STATE UNIVERSITY

DOE - \$108,328

6/1/77 - 5/30/80

Principal Investigator - R.C. Bradt

**OBJECTIVES** – This research seeks to investigate and understand the fracture characteristics of refractory concretes (castables) of the types under consideration as candidates for coal gasifier linings. These objectives will be pursued by experimentally measuring the fracture mechanics parameters necessary for coal gasifier design with refractory concrete linings. Generic refractory concretes and specially designed concretes will be studied from room temperature to 1200°C.

**RECENT WORK AND ACCOMPLISHMENTS** – Since the contract began in June 1977, two graduate research assistants have started on the project. Materials for the synthesis of the concretes have been ordered from commercial suppliers.

**PLANS FOR THE COMING YEAR** – Work will include synthesis of refractory concrete bodies and experimental measurements on the bodies.

## **TRANSITION METAL CLUSTER COMPLEXES AS CATALYSTS FOR CONVERTING COAL-DERIVED SYNTHESIS GAS INTO ORGANIC FEEDSTOCKS AND FUELS**

PENNSYLVANIA STATE UNIVERSITY

DOE - \$40,000; PSU - \$11,282

9/1/77 - 8/31/79

Principal Investigator - G.L. Geoffroy

**OBJECTIVES** – The overall goal of this research is to develop selective and efficient catalysts for the conversion of coal-derived synthesis gas into useful organic feedstocks and fuels. Work involves conducting a thorough evaluation of a variety of metal clusters for their ability to catalyze reduction of CO, optimizing the reaction conditions for those clusters identified as catalysts, and developing a thorough understanding of the interactions of CO and H<sub>2</sub> on transition metal clusters.

**RECENT WORK AND ACCOMPLISHMENTS** – Efforts have been devoted to establishing the reaction conditions and analytical procedures for evaluating the ability of specific clusters to catalyze reduction of CO. Each cluster will be initially screened under three different conditions:

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under the first set of conditions:  $\text{Fe}_3(\text{CO})_{12}$ ,  $\text{H}_2\text{FeRu}_3(\text{CO})_{13}$ ,  $\text{HCoRu}_3(\text{CO})_{13}$ ,  $\text{H}_4\text{Ru}_4(\text{CO})_{12}$ ,  $\text{CH}_3\text{CCo}_3(\text{CO})_9$ , and  $\text{PhCCo}_3(\text{CO})_9$ . None were found to catalyze reduction of CO under these conditions.

**PLANS FOR THE COMING YEAR** – The clusters previously mentioned will be evaluated under the second and third reaction conditions in the coming months. In addition, the following clusters will be screened under all three sets of conditions:  $\text{Ru}_3(\text{CO})_{12}$ ,  $\text{Os}_3(\text{CO})_{12}$ ,  $\text{Ir}_4(\text{CO})_{12}$ ,  $\text{Co}_4(\text{CO})_{12}$ ,  $\text{Rh}_4(\text{CO})_{12}$ ,  $\text{H}_3\text{Re}_3(\text{CO})_{12}$ ,  $\text{H}_2\text{Ru}_4(\text{CO})_{13}$ ,  $\text{Rh}_6(\text{CO})_{16}$ ,  $\text{HFeCo}_3(\text{CO})_{12}$ ,  $\text{H}_4\text{Os}_4(\text{CO})_{12}$ ,  $\text{Fe}_5\text{C}(\text{CO})_{15}$ , and  $\text{H}_3\text{Mn}_3(\text{CO})_{12}$ ,  $\text{Fe}_6\text{C}(\text{CO})_{16}^{2-}$ . Other clusters will be evaluated as they become available. Once clusters are found that catalyze the reaction, the reaction conditions will be optimized and the selectivities of the catalysts determined.

## RELATION OF COAL CHARACTERISTICS TO LIQUEFACTION BEHAVIOR

PENNSYLVANIA STATE UNIVERSITY

DOE - \$500,739; PSU - \$26,354

7/1/76 - 9/30/77

Principal Investigator - P.H. Given

**OBJECTIVES** – This country's large mass of coal reserves differ in rank, petrography, distribution of minerals and organic chemical structures, biological sources, conditions of metamorphism, and pore-size distribution. This study is seeking to determine whether, and in what ways, these and other characteristics of coals determine the yields of liquids and the composition of the products in coal liquefaction. The relationships found shall be expressed in forms that will aid the selection of coals for processes, the siting of plants, and also the selection of processing conditions for any given coal.

**RECENT WORK AND ACCOMPLISHMENTS** – A sample base of 110 coals has been selected for this project, all characterized as to chemical and petrographic analyses, and to the contents of inorganic species. The samples cover the range of rank from subbituminous to the upper end of the HVA class and represent most of the important coal-producing areas of the country. The selection from the Penn State/DOE Sample Base was made by computer. A new reactor system was constructed with which conversions have been determined in duplicate on 80 coals for their interaction with tetralin as donor solvent. In addition, one coal was run at two temperatures and five residence times to permit determination of the changes of the character of the liquid products and of the solid residue as functions of the level of conversion. The yield of higher molecular weight material (more or less equivalent to asphaltenes) was almost independent of the degree of conversion changes being largely confined to the lighter fraction. A study of mechanism and kinetics of coal-tetralin interactions is in progress, designed to throw light on important aspects of the phenomenology of the process. Conversion of a coal to pyridine-solubles, benzene-solubles, and hexane-solubles plus gas has been determined at five temperatures ( $340^\circ$  to  $400^\circ\text{C}$ ) and three to five residence times in the range 0 to 30 minutes. Loss of total oxygen and of phenolic hydroxyl content were also determined. No rapid initial increase of hydroxyl was observed, as would be expected if ether splitting is an important early step in liquefaction. At low levels of conversion, loss of total oxygen is faster than loss of phenolic OH, implying that some other form of oxygen is preferentially eliminated. The activation energy for the conversion of the coal to pyridine-solubles appears to vary somewhat with temperature and quite markedly with the level of conversion. The conventional separation of liquefaction products into oils and asphaltenes according to solubility in hexane and

benzene is not entirely satisfactory, because both polarity and molecular weight will determine solubility. A procedure has been developed for separating the crude products chromatographically on a gel with molecular sieve properties, so that only molecular size determines which substances report to one fraction or to the other. The heavier material is soluble only in pyridine or tetrahydrofuran, but after conversion of any phenols present to trimethylsilyl derivatives, the whole fraction becomes readily soluble in the non-polar solvent, carbon tetrachloride. This major change strongly suggests that before derivatization, there were important intermolecular interactions between phenols and other molecules.

**PLANS FOR THE COMING YEAR** – When liquefaction data for the full set of 110 subbituminous and bituminous coals are available, multiple regression analyses will be run to relate conversion to coal characteristics. A smaller set of coals will then be studied under SRC conditions to determine whether catalysis by mineral matter significantly affects the correlations found in the absence of hydrogen gas. In separate experiments, the effect of adding well-characterized known minerals to mixtures of demineralized coals with tetralin and hydrogen will be determined. Efforts to understand the phenomenology of liquefaction, including the role of the various minerals, will continue. The search will be carried on for fairly readily determined characteristics of liquefaction products that will permit product composition to be related to coal properties. Work on the nature of inorganic species in coals and on the structure of the organic matter will continue.

## INSTRUMENTAL ANALYSIS OF SULFUR COMPOUNDS IN COAL PROCESS STREAMS

PENNSYLVANIA STATE UNIVERSITY

DOE - \$136,397; PSU - \$7179

9/30/77 - 9/29/79

Principal Investigator - J. Jordan

**OBJECTIVES** – In-plant instrumental analysis methods are being developed for determining sulfur compounds in coal conversion process streams. This work will be accomplished by using basic kinetic and thermodynamic parameters to evolve two types of independent analytical procedures for the quantitative determination of each and every sulfur moiety identified as significant: an enthalpimetric method, based on the measurement of heats of reaction in an adiabatic cell; and a voltammetric method, using glassy carbon indicator electrodes. Precision, accuracy, and interferences will be assessed in simulated and actual coal conversion process stream samples. Enthalpimetric and voltammetric cells will be designed for performing in-plant analyses in “real time.”

**RECENT WORK AND ACCOMPLISHMENTS** – As a model “feasibility study,” the voltammetry

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profiles will be determined, specifying the moieties that exist in every oxidation state of sulfur ranging from -2 (e.g., sulfide) to +8 (persulfate). Based on this information, the "occurrence probability" of potential sulfur contaminants in coal conversion streams will be assessed. In the analytical procedures development, specificity will be optimized by choice of selective reagents for the calorimetric determinations and by judicious reliance upon controlled potential scans and intrinsically selective techniques (such as differential pulse measurements) in voltammetry. Sensitivity will be maximized by reliance upon appreciably exothermic or endothermic reactions and by the use of rotated disk electrodes.

## CHEMOMECHANICAL PHENOMENA IN THE GRINDING OF COAL

PENNSYLVANIA STATE UNIVERSITY

DOE - \$49,510; PSU - \$2606

2/1/76 - 5/31/77

Principal Investigator - N.H. Macmillan

**OBJECTIVES** – The aims of this program were twofold: first to determine whether coal exhibits the same sort of surface-charge controlled, chemisorption-induced variations in near-surface flow and flow-dependent fracture behavior (chemomechanical effects) as do inorganic rocks, minerals, ceramics, and glasses; and second, to endeavor to translate any such effects in coal into improvements in grinding efficiency.

**RECENT WORK AND ACCOMPLISHMENTS** – Hardness, drilling rate, grinding rate, and zeta potential measurements were made on a series of coals varying in rank from lignite to anthracite in two environments, one aqueous and one organic. The zeta potential studies were to provide an indication of the surface charge adopted by the coals in the different test environments, the Vickers microhardness studies would define chemomechanical effects that occurred, and the drilling and grinding rate studies would provide some indication of the practical value of these effects. These studies revealed only two negative results: first, that the mechanical properties of coals of any rank are much less environment-sensitive than those of inorganic rocks, minerals, ceramics, and glasses; and second, that such slight environment-sensitivity exhibited by the mechanical properties of coal apparently is not related to zeta potential. It was concluded, therefore, that coal does not exhibit any obviously predictable and useful chemomechanical effects, and that there appears to be little prospect of reducing the cost of grinding coal through the use of chemisorption-induced changes in its near-surface flow and/or flow-dependent fracture properties. The work did demonstrate that the mechanical behavior of coal is not entirely environment-insensitive. Specifically, methanol and aqueous sodium sulfonate solutions were found to lower by ~20 percent and butanol to raise by ~50 percent the hardness of the (dominant) vitrite microlithotype in a high-volatile bituminous A coal. However, none of these environments, nor any other environment studied produced any effect on drilling rate large enough to detect against background experimental scatter. And the only effect of environment apparent on overall grinding performance, as indicated by the Hardgrove Grindability Index (HGI), was a slight increase in the case of anthracite in the (more viscous) longer chain alcohols and slight decreases in the cases of certain lower-rank coals in these same environments. Moreover, this almost complete indifference of overall grinding behavior to environment was also reflected in the similarity of the size distributions of the fines produced during these HGI determinations, although there was some inconclusive evidence that this indifference was derived from the mutual cancellation of the opposing effects of different environments on the primary daughter fragment distribution function and on the specific rate of breakage parameter.



**PLANS FOR THE COMING YEAR** — There are no plans to pursue these studies further since they appear to offer little prospect of any payoff.

### STABILITY OF $\text{SiC}$ , $\text{Si}_3\text{N}_4$ , $\text{Si}_2\text{N}_2\text{O}$ AND SIALON IN COAL GASIFICATION ENVIRONMENTS

PENNSYLVANIA STATE UNIVERSITY

DOE \$202,779; PSU - \$10,672

9/1/77 - 8/31/80

Principal Investigator - A. Muan

**OBJECTIVES** — This research is concerned with the chemistry of carbide-nitride-oxynitride refractory phases in slagging coal gasifier environments. The specific objectives are: (1) to evaluate stability relations of  $\text{SiC}$ ,  $\text{Si}_3\text{N}_4$ ,  $\text{Si}_2\text{N}_2\text{O}$ , and Sialon as a function of oxygen-carbon-nitrogen fugacities in the temperature range of approximately 2000° to 3000°F; (2) to evaluate the effects of various slags on the stabilities of these phases in atmospheres similar to those encountered in coal gasification processes; (3) to determine the rates of reaction between these phases and slag phases similar to those encountered in coal gasification processes; and (4) to ascertain the maximum temperatures at which commercial silicon carbide refractories are stable in slagging coal gasifier environments.

**RECENT WORK AND ACCOMPLISHMENTS** — Main efforts have been exerted on building, modifying, and calibrating furnaces and other auxiliary equipment with a view toward optimizing their efficiency for studies of stability relations among phases in the system Si-Al-C-N-O.

**PLANS FOR THE COMING YEAR** — It is anticipated that the work during the coming year will be concentrated in two main areas, one involving the determination of stabilities of phases in the system Si-Al-C-N-O under atmospheric conditions approximating those prevailing in coal gasifiers, which will be accomplished by carrying out equilibrium measurements in a gas phase of known chemical potentials of oxygen, nitrogen, carbon, hydrogen, and water; and the other the determination of interactions between the previously mentioned refractories and various slags. Available equilibrium data on oxides will be used to estimate theoretically the probable interactions between refractory and slag. Key experiments will then be carried out to confirm or refute results of the calculations and to establish experimentally the main trends and mechanisms of interactions between the carbide-nitride-oxynitride phases on the one hand and the oxide (slag) phases on the other hand.

### REFRACTORY EROSION IN COAL GASIFICATION AND COMBUSTION SYSTEMS

PENNSYLVANIA STATE UNIVERSITY

DOE - \$40,000; PSU - \$2100

9/1/77 - 8/31/79

Principal Investigator - H.E. Shull

**OBJECTIVES** — Two complementary tasks are to be carried out to gain understanding of the high-temperature erosion resistance of alumina refractories in coal gasification and in advanced coal combustion environments. Using data generated from an experimental study of the erosion behavior of two selected aluminas under jet impingement conditions as a function of temperature and of impinging particle size, velocity, strength (hardness), and direction, the best existing theory of the erosion of brittle materials will be tested. Simultaneously, an investigation will be conducted on these same materials of the potential of laser holography as a method of studying single-particle

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impacts in their various stages. By this method, the particle impact velocity data needed in the former study will also be provided.

**RECENT WORK AND ACCOMPLISHMENTS** – The initial months of this research have been dedicated to restoring the laser holographic system to proper operating condition. The Materials Research Laboratory's 10 joule double-pulse Korad holographic system has been disassembled, cleaned, reassembled, and aligned, after an extended period of non-use. Essential diagnostic optical and electronic equipment has been ordered to permit reliable operation of the system.

**PLANS FOR THE COMING YEAR** – The initial year's experiments will be limited to room temperature, while the design and construction of an appropriate furnace system is being carried out. Target materials will be prepared and subjected to jet impingement erosion designed to simulate the low particle concentration/high particle end of the spectrum of operating conditions found in typical gasifiers and in advanced coal combustion systems. Detailed investigations will be made of the size and shape of the spent erosive particles, of target debris, and of the worn target surfaces. Pulsed laser holography will be evaluated as a method of measuring particle velocities, and used as such if feasible. At the same time, efforts will be applied to develop holography as a practical tool for examining single-particle impacts.

## CHARACTERISTICS OF AMERICAN COALS IN CONVERSION INTO CLEAN ENERGY FUELS

PENNSYLVANIA STATE UNIVERSITY

DOE - \$3,220,894; PSU \$345,633

6/26/75 - 8/25/79

Principal Investigator - W. Spackman

**OBJECTIVES** – The overall goal is the prediction of the response of coal in beneficiation, gasification, liquefaction, and combustion processes. Specific objectives of the program include sampling and detailed characterization of coals, covering the spectrum of the Nation's major coal reserves; investigation of the preparation characteristics of U.S. coals and of the feasibility of manipulating coal composition for conversion processes; design, construction, evaluation, and use of bench-scale reactors and reactivity tests; development of an understanding of the factors that affect the structure of chars produced by the thermal treatment of coals of varying rank and relating variability in char structure to char reactivity in different atmospheres; and determination of the requirements for the use of low-volatile coals and chars in pulverized coal boilers.

**RECENT WORK AND ACCOMPLISHMENTS** – This year 368 coal samples have been obtained, bringing the total number of samples collected during the course of the program to 631. In addition to the basic petrographic, chemical, and physical characterization, an analytical scheme has been developed for the determination of most major, minor, and trace elements and mineral species; approximately one third of the proposed data has been collected. A unique coal fractionation scheme permits development of coal products having strikingly different compositions. Studies are establishing the extent to which such products result from commercial preparation operations and investigating the feasibility of enhancing this compositional control. Pneumatic (Dry Flo separator) and wet phase (oil agglomeration) fine coal beneficiation techniques are being studied. Experimental evidence obtained on four lignites and on a high-volatile bituminous coal suggests two-stage mechanisms of pyrolysis consistent with certain gross models of coal constitution. Reactivity of coal and coke in nonpressurized gasifying conditions, examined in a combustion pot, has shown

that in the combustion section of the bed, reactivity is boundary-layer diffusion controlled, while in the gasification section it depends on the kinetic behavior of the materials.

Other work involves techniques for the characterization of chars using measurement of char density, surface area, and pore volume: altering char structure by either preoxidation or inorganic addition to precursor coals; correlating char reactivity with char structure and catalytic activity of various inorganic additives; and characterizing the interaction of oxygen with active sites in chars by measuring heats of interaction. All coal char-reactant gas combinations have been unified into one reactivity plot with a high degree of correlation. The only adjustable parameter required is time to reach a fractional burnoff of 0.5. This unification means that there is a commonality in char gasification related to the opening up of closed porosity, the enlargement of open pores, and finally the total consumption of pore walls and, thus, a decrease in number of pores. The rate of evolution of pore structure is a function of reactant gas used, but the character of evolution is essentially independent of reactant gas. It has also been demonstrated that reactivity of lignite chars can be dramatically increased concurrent with increase in calcium content of the precursor lignite. Char reactivity to steam was increased by a factor of greater than 10. A study of the combustion of chars has shown that they can range in reactivity from values close to those for anthracite up to values found for bituminous coal. Reactivity appears dependent not on volatile matter, per se, but on internal surface area and its accessibility, which are determined by pretreatment. Particle size is



*Point-Count Analysis of Maceral Composition of Coal*

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important for low-reactivity chars and anthracite, but for high-reactivity chars behavior is insensitive to particle size. Coal in amounts up to 15 percent have been added to oil-water-air emulsions to determine heat transfer and combustion characteristics. A peaking of thermal efficiency at 6 to 7 percent coal additions is attributed to differential contributions to heat transfer from the flame and from the hot roof and walls.

**PLANS FOR THE COMING YEAR** — The evaluation of the beneficiation characteristics of coals will be extended to include more commercial-plant relationships. A new laminar-flow isothermal reactor will be commissioned to investigate the effects of pressure on pyrolysis in various gases. Emphasis will be applied to gaining a better understanding of the phenomenon of why coal preoxidation affects coal plasticity, subsequent char structure, and reactivity; evaluating the phenomenon of metal carbonate addition to coal offsetting its plasticity and subsequent char reactivity; developing quantitative relationships between the change in concentration of active carbon sites and reactivity with carbon burnoff during gasification in air; relating heats of oxygen transfer to coal chars to their gasification propensity in air; and using small-angle x-ray scattering to augment the characterization of the pore structure in chars. Additional chars will be acquired from DOE pilot plants for further flame experiments involving determination of gas and solids concentrations.

## STATE OF THE ART OF POWER PLANT CONSTRUCTION PROGRAM

PENNSYLVANIA STATE UNIVERSITY

DOE - \$51,065

4/1/76 - 6/30/77

Principal Investigator - J. Willenbrock

**OBJECTIVES** — The purpose of this program was to provide a mechanism for "technology transfer" from industry to the classroom that would assist engineering faculty in the development of curriculum material related to the construction of both fossil and nuclear power plants. Secondly, it was hoped to build a bridge of communication between engineering faculty involved in construction education and their counterparts in the power-plant construction industry that would lead to future cooperative educational and research efforts.

**RECENT WORK AND ACCOMPLISHMENTS** — A 2-week seminar entitled "The State of the Art of Power Plant Construction," dealing with both fossil and nuclear facilities, was developed and presented in August 1976 at the Pennsylvania State University. The objective of this course was to "educate" engineering faculty in a better understanding of the construction process required to build complex long-duration power plants. The primary source for lecturers for this conference was the representative companies in the power-plant construction field. The seminar was attended by 43 educators and 34 industry representatives. A 2-day "Educational and Research Needs" planning meeting was held at Penn State in May 1977 that built upon and reinforced the educational partnership started at the August 1976 seminar between the academic community and industry and established plans for a mutual effort that would satisfy both the educational as well as the research needs in the power plant construction industry. The attendees at this meeting (22 educators and 34 industry representatives) included high-management-level personnel who had an overview of the educational and research needs of the industry, and who were willing to share their views about these topics. They had the opportunity to interact with engineering faculty members from U.S. schools who were teaching construction engineering and construction management programs.

**PLANS FOR THE COMING YEAR** — This project was completed in June 1977.

## KINETICS AND MODELING OXIDATIVE PRETREATMENT OF COAL

UNIVERSITY OF PENNSYLVANIA

DOE - \$63,425; UP - \$6207

7/1/77 - 6/30/78

Principal Investigator - D.D. Perlmutter

**OBJECTIVES** – A major processing difficulty arises in coal conversion from the tendency of common coals to soften and agglomerate when heated to process temperatures. One method of handling this problem uses oxidation at moderate temperatures to pretreat the coal. This study is experimentally investigating the kinetics of this pretreatment to relate the data to proposed interpretive models and to correlate the kinetic data with the changes in coal properties. Seven coals from different sources are being tested at various temperatures and pressures in fixed-bed reactors under a range of flow rates and for various particle sizes. The coals pretreated in this manner will be evaluated by a gas-flow caking test for correlation to the oxidative changes.

**RECENT WORK AND ACCOMPLISHMENTS** – The most striking single result is the finding that the coals tested may be classified in two categories, according to whether or not the rate of oxidation monotonically falls during the 4 to 7 hours of a test. By listing the coals in the order of their pore volumes of relatively large diameter (greater than 300 Å), they can conveniently be divided into two subsets. The behavior patterns of the small-pore coals conform to the essential features of the Kam-Hixson-Perlmutter (KHP) model (1976), as regards both kinetics and a mass transfer boundary layer effect. The ratios of CO<sub>2</sub> and CO produced were computed and found to be independent of particle size and conversion. The samples of lignite and a high-volatile C bituminous coal oxidized in this study had appreciably larger pores and greater chemical activity than the other samples. The results show extended periods of high activity during which the rates were determined by the limiting oxygen reactant. Measured rates dropped as the most reactive surface or pore-available sites were used up and diffusional mechanisms began to play a major role. The rates fell sharply when larger particles, reduced temperatures, or reduced flow rates were used.

**PLANS FOR THE COMING YEAR** – Work will focus on completing the range of test variables and on an assessment of the process by which CO<sub>2</sub>, CO, and water are produced. Caking tests and coal analyses on exposed samples are yet to be run and formal models and correlations developed.

## HYDROGEN DISTRIBUTION AND TRANSFER IN COAL HYDROGENATION SYSTEMS

UNIVERSITY OF PITTSBURGH

DOE - \$110,431; UP - \$44,493

9/1/77 - 8/31/79

Principal Investigator - S.H. Chiang

**OBJECTIVES** – This research project is investigating the behavior of hydrogen in coal hydrogenation systems to provide basic data for optimum design of coal liquefaction processes. It is designed to achieve the following goals: to design, construct, and demonstrate a nickel-membrane hydrogen probe for measuring hydrogen partial pressure and concentration in hydrogen/oil/coal and simpler system(s); to obtain equilibrium (steady-state) hydrogen distribution—in both elemental and chemically combined forms—in coal hydrogenation systems; and to study the dynamic (unsteady-state or transient) behavior of hydrogen transfer between gas phase and condensed phase (liquid/solid mixture) in the system(s).

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**RECENT WORK AND ACCOMPLISHMENTS** — The initial phase of the project has been devoted to the design, construction, and calibration of a hydrogenation unit and Ni-membrane hydrogen probes. The hydrogenation unit consists of a high-pressure autoclave (rated at 5000 psi working pressure), a high-pressure gas manifold, a temperature controller, precision pressure gauges, and gas/liquid samplers. The hydrogen probe is made of 1/8-in. stainless steel (300 series) tubing with a thin, nickel, semipermeable membrane tip. The thin Ni-membrane, supported by inert packing materials, can be adapted for high-pressure services. The design/construction phase of the project is expected to be completed by December 1977.

**PLANS FOR THE COMING YEAR** — Work for the coming year will include: testing and calibrating the Ni-membrane hydrogen probes; determining equilibrium hydrogen distribution in coal-derived recycle oil (Synthoil, for example); and determining the equilibrium hydrogen distribution in mixtures containing hydrogen, oil (coal-derived recycle oil) and coal.

## HOT CORROSIVITY OF COAL GASIFICATION PRODUCTS ON GAS TURBINE ALLOYS

UNIVERSITY OF PITTSBURGH

DOE - \$143,994

Principal Investigators - G.H. Meier, R.A. Stoehr

**OBJECTIVES** — This program seeks to develop information about the hot corrosion of gas turbine alloys in the environment likely to be found when a gas turbine is operated on low-Btu gas produced from coal in a fluidized-bed gasifier. It is designed to determine the mechanisms of attack and the major factors influencing the kinetics of hot corrosion in these environments. Existing gas turbine alloys and a few simple alloys of potential gas turbine alloy composition have been selected for inclusion to be representative of several different metallurgical types exhibiting different hot corrosion behavior.

**RECENT WORK AND ACCOMPLISHMENTS** — The scope of the program includes: (1) gathering of data to ascertain the range of environmental conditions of importance in a turbine-burning gasified coal; (2) thermochemical calculations to determine the important condensed-phase and vapor species to be expected in a turbine; (3) study of reactions between condensed-phase deposit mixtures and the oxides that form on turbine alloys; (4) simple high-temperature oxidation experiments on the alloys of interest; and (5) hot corrosion experiments on the same alloys using the deposits which (1) and (2) indicate to be the most important. The results of (1) have indicated that  $\text{Na}_2\text{SO}_4$ ,  $\text{K}_2\text{SO}_4$ , and their mixtures should be the primary detrimental condensed phases in the temperature range of interest (1150° to 1450°K). In (2), the existing thermochemical data have been evaluated and metal-sulfur-oxygen condensed phase stability diagrams have been constructed at 1150°, 1250°, 1342°, and 1450°K for the metals Na, K, Mg, Ca, Al, Co, Cr, Mo, Ni, Si, Ti, V, and W. Results of crucible tests in (3) have shown that  $\text{Al}_2\text{O}_3$  does not react appreciably in air with pure liquid  $\text{Na}_2\text{SO}_4$  or a mixture of  $\text{Na}_2\text{SO}_4$  and  $\text{K}_2\text{SO}_4$ . However, addition of MgO or  $\text{MgSO}_4$  to the  $\text{Na}_2\text{SO}_4$  results in the formation of  $\text{MgAl}_2\text{O}_4$  as a reaction product. The results of (4) and (5) obtained on pure Ni and Co, at oxygen pressures of 0.2 and  $10^{-6}$  atm have shown that liquid deposits of  $\text{Na}_2\text{SO}_4$  result in accelerated corrosion. Results of optical and scanning electron metallography and x-ray analysis of the corroded specimens have indicated a mechanism whereby an initial oxide film is broken down and allows  $\text{Na}_2\text{SO}_4$  to penetrate under the scale. Sulfides formed during the initial stages are oxidized when the scale is penetrated. The repetition of this process results in the formation of a honeycomblike scale. Hot corrosion experiments using several commer-

cial Ni- and Co-base alloys and simple alloys such as Ni-6Al, Ni-20Cr, Ni-10Cr-4Al, Ni-50Cr, Co-20Cr, Co-30Cr, Co-7.5Ti, and Co-20Cr-7.5Ti have indicated that simple binary Ni-20Cr and Co-30Cr alloys perform better than the more complex alloys in exposures using pure  $\text{Na}_2\text{SO}_4$  and  $\text{Na}_2\text{SO}_4$  modified with  $\text{K}_2\text{SO}_4$  or  $\text{SiO}_2$ . The addition of third elements, particularly those added for solid solution strengthening, generally degrade the hot corrosion resistance of the simple alloys. The results also show that  $\text{K}_2\text{SO}_4$ , when added to  $\text{Na}_2\text{SO}_4$ , results in extremely severe attack of some alloys (e.g., IN-738) that have good resistance in pure  $\text{Na}_2\text{SO}_4$ . Additions of  $\text{SiO}_2$  also result in severe corrosion of many of the alloys.

**PLANS FOR THE COMING YEAR** – The study of the mechanism of hot corrosion of alloys using a wide range of salt deposits will be continued and intensified. Particular emphasis will be given to the effects of  $\text{K}_2\text{SO}_4$ ,  $\text{SiO}_2$ , and carbon. Also, the hot corrosion of several new alloys will be studied. Experiments whereby the gas phase is doped with  $\text{SO}_2$ ,  $\text{H}_2\text{S}$ , and  $\text{CH}_4$  are beginning and will be continued. Further thermochemical calculations involving vapor species are also planned.

### ANALYTICAL MODELING OF PRACTICAL COMBUSTION SYSTEMS

CLEMSON UNIVERSITY  
DOE - \$25,461; CU - \$1492  
12/01/76 - 9/15/78  
Principal Investigator - G.F. Robinson

**OBJECTIVES** – This program intends to provide a validation test for one or more existing two-dimensional, recirculating-flow, combustion models to make an experimental evaluation of the various constants that are contained in these models and to select the most fruitful direction for further development work.

**RECENT WORK AND ACCOMPLISHMENTS** – Work completed includes the detailed study of a two-dimensional, recirculating-flow, combustion model developed by Combustion, Heat, and Mass Transfer, Ltd. (Wimbledon, United Kingdom). This model is a general one designed to treat many classes of flow: steady or unsteady, laminar or turbulent, incompressible or compressible, non-reacting or reacting, and with or without radiative heat transfer. Turbulence is treated by a two-equation turbulence model. The effect of turbulence on the chemical reaction rate is treated by Spalding's Eddy Breakup Model or alternative methods. The partial modification of the overall model and the initial implementation of the revised model were completed.

**PLANS FOR THE COMING YEAR** – This analytical model will be applied to a simple burner that previously furnished experimental data. These data have been obtained over a range of turbulence conditions, and, consequently, a particular focus of the work will be an evaluation of the model's ability to predict experimentally observed trends that appear as turbulence conditions. The model's ability to describe the interplay between turbulence and chemical kinetics will also be investigated as will the sensitivity of the model to uncertain parameters and boundary values.



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## FOSSIL ENERGY RESEARCH PARTICIPATION AND FACULTY ROTATOR PROGRAMS

### OAK RIDGE ASSOCIATED UNIVERSITIES

DOE - \$292,000

4/1/76 - Continuing

Principal Investigator - A. Wohlpart

**OBJECTIVES** – The Fossil Energy Research Participation Program seeks to obtain the services and expertise of university faculty members and graduate students through their participation in the ongoing R&D activities at the DOE Energy Research Centers in Bartlesville (Oklahoma), Morgantown (West Virginia), and Pittsburgh (Pennsylvania). At the same time, such research participants will be trained in fossil energy process technology. Faculty members will be in an exceptional position to incorporate this knowledge into their own research programs and into the educational process. The objective of the Faculty Rotator Program is to secure the assistance and experience of faculty members through their participation in program planning and review in the Fossil Energy Headquarters in Washington. Appointments for faculty members will generally be for the summer months; a limited number of longer appointments may be made in instances where a faculty member combines the appointment with a sabbatical. Appointments for graduate students will either be for the summer or for a sufficient length to permit the completion of a thesis or dissertation research project.

**RECENT WORK AND ACCOMPLISHMENTS** – Announcements for these programs are routinely sent to more than 6000 science and engineering faculty members at more than 400 U.S. colleges and universities. In 1976 and 1977, nearly 100 applications were received. Since 1976, 41 faculty members and 2 graduate students have participated in the work of the DOE facilities cited above, for a total of 7.3 and 1.0 man-years of performance, respectively. Participants, immediately prior to completion of their appointments, submit to ORAU and the host center a report of the summer research activities. Participants have published one paper and given six talks as a result of their research participation.

**PLANS FOR THE COMING YEAR** – By the end of December 1977, a brochure describing the qualifications, opportunities, and terms that apply to the DOE-Fossil Energy Programs will have been prepared and mailed to approximately 5000 science department chairmen and to 1000 individuals. Applications will be received and assembled in January and transmitted to the designated Energy Research Center or appropriate Washington Office by mid-February for selection of applicants in collaboration with ORAU. ORAU intends to notify successful candidates by mid-March.

## MOLECULAR WEIGHT DISTRIBUTIONS OF COAL AND CONSTITUENT MOLECULES

### UNIVERSITY OF TENNESSEE

DOE - \$100,000

3/1/77 - 2/28/79

Principal Investigator - J.W. Larsen

**OBJECTIVES** – This project seeks to determine the molecular weight distributions of coals and of the molecules that are polymerized to form the coal macromolecules. Many of the chemical and physical properties of polymers are controlled by their molecular weight distribution, cross-link density, and monomer size, which is true of coal. It is planned to determine whether these macro-



molecular characteristics can be used to predict the behavior of various coals in liquefaction processes.

**RECENT WORK AND ACCOMPLISHMENTS** – It has been demonstrated that the plastic properties of coals can be used to discover their macromolecular structure. Applying these techniques to bituminous coals shows that they are three dimensionally cross-linked macromolecular gels having a number average molecular weight per cross link of 1500 to 1800. Bruceton coal has been successfully depolymerized using the Heredy-Neuworth technique and the product separated using size exclusion high-pressure liquid chromatography. The products seem to have a rather narrow molecular weight distribution, number average molecular weight - 325. Reductive alkylation of Illinois No. 6 coal has been carried out successfully and product analyses are underway.

**PLANS FOR THE COMING YEAR** – Synthesis of radioactive naphthalene and tetrahydrofuran will be completed and these  $^{14}\text{C}$  labeled compounds will be used in the reductive alkylation of several coals. More coals and some pure molecules will be depolymerized and the molecular weight distributions obtained and compared with their behavior in liquefaction processes.

### CATALYSTS FOR COAL LIQUEFACTION

UNIVERSITY OF TENNESSEE  
DOE - \$64,475  
6/1/75 - 11/30/77  
Principal Investigator - J.W. Larsen

**OBJECTIVES** – This project intends to investigate whether any known catalyst systems are capable of causing coal liquefaction under very mild conditions. If no such systems exist, the objective is to develop one. Coal liquefaction processes require high temperatures and pressures, which cause enormously expensive plants. Coal is a very reactive material, indeed the difficulty in liquefying it probably stems from too high a reactivity rather than too low. A catalytic system capable of causing coal liquefaction under mild conditions would have enormous advantages over all current processes.

**RECENT WORK AND ACCOMPLISHMENTS** – Catalyst systems that might be effective in coal liquefaction, judging from experience with pure compounds, were identified and tested. None are capable of coal liquefaction under mild conditions. A number of new systems were tried, and one very promising reaction was developed. Ionic hydrogenation using  $\text{BF}_3 \cdot \text{H}_2\text{O}$  and triethylsilane is capable of selectively removing sulfur and nitrogen from model polycyclic aromatics. Half of the hydrogen introduced comes from water, the reactions occur at room temperature, and take ca 1 to 2 hours to complete. This reagent removes ca 50 percent of the sulfur and ca 50 percent of the nitrogen from bituminous coals at  $25^\circ\text{C}$ . While a commercial process would have to use a cheaper hydride than triethylsilane, this reaction clearly establishes the selectivity of ionic hydrogenation and demonstrates that further investigation of this route to coal liquids is worthwhile.

**PLANS FOR THE COMING YEAR** – Experimental work has been terminated, and the final report is in preparation.

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## PHASE EQUILIBRIA IN COAL HYDROGENATION SYSTEMS

WILLIAM MARSH RICE UNIVERSITY

DOE - \$247,973; RU - \$14,739

4/1/76 - 3/31/79

Principal Investigator - R. Kobayashi

**OBJECTIVES** – This project intends to provide reliable thermodynamic data for mixtures of organic compounds at temperatures and pressures encountered in coal liquefaction and SRC process streams. In particular, this research is concerned with the measurement of vapor-liquid equilibria and hydrogen solubility in multicomponent poly- and heterocyclic systems at pressures up to 4000 psia and at temperatures from ambient to 700°F. Completion of these studies will provide fundamental information crucial to process development and equipment design of coal liquefaction and solvent extraction plants.

**RECENT WORK AND ACCOMPLISHMENTS** – Construction has been completed on most of the two experimental systems designed for this research. The enclosed high-temperature baths, consisting of nitrogen-blanketed fluid tanks, external heating elements, and a surrounding insulation housing, is raised and lowered hydraulically underneath a fixed instrumentation platform. This platform is contained within a protective fume hood. The perturbation chromatography system that will be used to measure K-values incorporates a low-temperature trap and a diaphragm pressure indicator to minimize problems associated with high component volatility at the temperature being studied. The elution gas is presaturated with the material under study so that a steady-state approximation to equilibrium can be assumed. The diaphragm indicator allows continuous monitoring of pressure without having material condense in the room-temperature regions of the system. All wetted components have been fabricated from A-286SS or 316 SS to minimize the dangers associated with hydrogen embrittlement. A combination equilibrium cell and precision transfer pump to be used for the measurement of the solubility of H<sub>2</sub> and associated gases in coal components has been constructed. The chromatographic subsystem for this equipment has been assembled and tested. A 17cc micropump for precise metering of organic compounds at elevated temperatures has also been completed.

**PLANS FOR THE COMING YEAR** – Work will include the compilation and correlation of data for well-defined thermodynamic systems of interest. Preliminary experiments on the perturbation chromatography equipment are expected to begin during the first quarter of 1978.

## COAL LIQUEFACTION AND DESULFURIZATION IN MULTIPHENOLS

UNIVERSITY OF HOUSTON

DOE - \$40,000; UH - \$1605

9/1/77 - 8/31/79

Principal Investigator - A. Attar

**OBJECTIVES** – The project intends to develop preliminary data on the feasibility of the use of multiphenols (MP) for coal liquefaction. It has been demonstrated that MP can be used to liquefy coal at relatively low temperatures and pressures. Experimental work will obtain data on liquefaction efficiency, kinetics of the desulfurization, and feasibility of solvent recovery to perform an economic evaluation of the proposed low-pressure liquefaction process.

**RECENT WORK AND ACCOMPLISHMENTS** – The kinetics of the desulfurization of Texas lignite, Illinois No. 6, Kentucky No. 9, Monterey coal, Amax coal, and coal from the Pittsburgh seam were examined. Most of the organic sulfur and the pyritic sulfur were desulfurized below 400°C and 100 psi. An optimal rate of heating exists, which if exceeded, prevents all the pyritic sulfur from being removed. The rate of depyritization seems to depend on the size distribution of the pyrite particles. The rate of desulfurization of the organic sulfur depends on the distribution of organic sulfur functional groups in the coal and on the solvent. Preliminary tests were conducted to evaluate liquefaction efficiency. Analytical methods are being studied that will permit a quantitative evaluation of the products of the liquefaction.

**PLANS FOR THE COMING YEAR** – A systematic study of the kinetics of the desulfurization and of the rate of liquefaction of one coal will be conducted. The distribution of the molecular weights of the liquefaction products will be determined by gel permeation chromatography, and the data will be correlated with the reaction conditions. NMR, IR, and UV will be used to evaluate the liquefaction products.

#### HEAT SUPPLY TO FLUIDIZED BED BY PARTICULATE HEAT TRANSFER

UNIVERSITY OF HOUSTON  
DOE - \$97,822; UH - \$3485  
8/77 - 8/79  
Principal Investigator - R. Jackson

**OBJECTIVES** – This work seeks to investigate heat transfer to a fluidized bed from larger or denser particles continuously passed downwards through the bed and removed from below. This method of heat supply can be used in endothermic reactions such as in the steam gasification of coal. Concurrent feed of air or oxygen is then unnecessary.

**RECENT WORK AND ACCOMPLISHMENTS** – The first phase of the work will identify quantitatively those factors which determine the holdup of the descending particles in terms of their flow rate and will determine the upper limits for the flow of large particles to be fed to the surface of a 4-inch-diameter fluidized bed of the smaller particles. After preliminary experimental work on the feed system, the main test rig is now under construction. The bed itself, the particle collection boot, the air supply system, and the feed and collection hoppers are complete and installed. The particle flow regulators and the weighing system are still to be completed.

**PLANS FOR THE COMING YEAR** – After completion of the test rig early in 1978, measurements of flow rate and holdup descending particles will be carried out. Designs will also be prepared for modifications to the rig to permit the heat transfer measurements. Construction of the heat transfer equipment will be initiated. It is also planned to develop a semitheoretical model of the mechanism of descent of the large particles through the fluidized bed.

#### CONVERSION OF COAL-BASED METHANOL TO ETHYLENE

TEXAS A&M RESEARCH FOUNDATION  
DOE/NSF-RANN - \$100,000; Texas A&M - \$5562  
10/1/75 - 9/30/77  
Principal Investigator - R.G. Anthony

**OBJECTIVES** – This project will determine the technical and economic feasibility of converting coal-based methanol to ethylene and to a gaseous fuel. The major emphasis of the experimental

work was to develop a catalyst to convert coal-based methanol to ethylene, because there was no known catalyst that would selectively convert methanol to ethylene or even to low molecular weight olefins.

**RECENT WORK AND ACCOMPLISHMENTS** — The gaseous fuel mixture, composed of 50 percent dimethyl ether, 33.3 percent hydrogen, and 16.7 percent carbon monoxide, has a heat of combustion and flame temperature approximately equal to that of natural gas. Hence, it offers an attractive alternate for conversion of methanol to a gaseous fuel similar to natural gas as opposed to conversion to synthetic natural gas (SNG-methane). A plant to produce 100 million ft<sup>3</sup>/d of gas mixture measured at 60°F and 1 atm, was designed, and the cost of producing the gas mixture was estimated. The conversion of methanol to ethylene involved testing several catalysts with varying amounts of tungsten oxide and with different ratios of silica-alumina in an attempt to improve the selectivity of the catalyst. Dimethyl ether was also passed over the catalyst to determine if it would be converted to ethylene. After a 2-hr induction period, ethylene and low-molecular weight olefins were produced. A major difficulty with the tungsten oxide catalyst was the formation of tungsten carbonyl which vaporized and then condensed in the tubing leaving the reactor, thus plugging the reactor effluent. The small pore zeolite, type 5A, was tested, and increased yields of ethylene at reaction temperatures of 350° to 450°C and liquid hourly space velocities of 0.1 to 0.5 were obtained. AW500 was then tested, and even better yields were obtained. The AW500 was then exchanged with rare earths, and the yields increased. The AW500 exchanged with rare earths is the best catalyst obtained to date.

**PLANS FOR THE COMING YEAR** — Work under the present project has been completed.

## **RHEOLOGICAL PROPERTIES OF METHACOAL SUSPENSIONS FROM TEXAS LIGNITES**

TEXAS A&M RESEARCH FOUNDATION

DOE - \$35,778; Texas A&M - \$9242

9/1/77 - 8/31/78

Principal Investigator - R. Darby

**OBJECTIVES** — This project seeks to evaluate the viscous flow rheological properties of suspensions of lignite coal in methyl alcohol, referred to as methacoal. Apparent viscosity data as a function of shear rate are to be obtained over a wide range of shear rates for a variety of methacoal suspensions. In addition to these rheological studies, a tube flow apparatus will be constructed for measuring pressure drop as a function of flow rate for such suspensions.

**RECENT WORK AND ACCOMPLISHMENTS** — Approximately 60 samples of methacoal suspensions have been formulated in a laboratory blender using lignite with initial moisture content ranging from 0 to 15 percent by weight. A variety of blending conditions were used which produced suspensions of varying particle size distributions and varying degrees of stability. After these data have been obtained, they will be analyzed to determine the non-Newtonian model that best represents the viscosity functions. The model parameters will then be correlated with solids concentration, particle size distribution, and lignite moisture content. In addition, construction of the tube flow apparatus has been started.

**PLANS FOR THE COMING YEAR** — The project work will be completed this year.

## FUNDAMENTAL ORGANIC CHEMISTRY OF COAL

TEXAS A&M RESEARCH FOUNDATION

DOE - \$39,942; Texas A&M - \$3618

9/1/77 - 8/31/79

Principal Investigator - C.S. Giam

**OBJECTIVES** – Novel organic chemistry procedures and reactions will be used to mildly, efficiently, and specifically degrade the “coal polymer” into smaller molecular fragments. Elucidation of the structure of these fragments should provide a valuable key to the coal structure itself.

**RECENT WORK AND ACCOMPLISHMENTS** – Derivatization and depolymerization of Texas lignites are being studied using novel mixed sulfonic-carboxylic anhydrides. Initial results indicate solubility enhancement under conditions which are milder than traditional ones. Because lignin is a major component of wood, it is probable that remnants of the lignin polymer remain intact in coal, especially in younger coals (lignite). A site specific and highly selective lignin “unraveling” scheme involving reaction with thioacetic acid-boron trifluoride, followed by Raney nickel desulfurization, is being applied to lignite degradation studies.

**PLANS FOR THE COMING YEAR** – Investigation will continue into the use of novel derivatization-depolymerization procedures. Reaction conditions will be optimized, and structural elucidation of coal fragments using column chromatography, infrared, ultraviolet, and nuclear magnetic resonance spectroscopy, as well as gas chromatography-mass spectrometry will begin. Initial studies will be carried out on Texas lignites. Eventually, the procedures developed will be applied to lignites from other states as well as to coals of all rank.

## TRANSITION METAL-GRAPHITE CATALYSTS FOR PRODUCTION OF LIGHT HYDROCARBONS FROM SYNTHESIS GAS

TEXAS A&M UNIVERSITY

DOE - \$87,918; Texas A&M - \$4676

8/1/76 - 7/31/79

Principal Investigator - M.P. Rosynek

**OBJECTIVES** – In view of the increasing amounts of carbon monoxide that will become available from the gasification of coal, this project was undertaken to develop a novel process for the production of petrochemical feedstocks based on coal or on other carbonaceous materials. Specifically, the research seeks to investigate the catalytic activities and selectivities of novel alkali and transition metal-graphite intercalates in producing light ( $C_1$ - $C_3$ ) hydrocarbons from  $H_2/CO$  synthesis gas via the Fischer-Tropsch process.

**RECENT WORK AND ACCOMPLISHMENTS** – A controlled-atmosphere chamber has been constructed for the handling of alkali metals and transition metal chlorides that must be protected from atmospheric water and oxygen during the initial steps of the catalyst preparation technique. An atmospheric-pressure, recirculation-type batch reactor system, with associated product analysis instrumentation, has been designed and fabricated to permit rapid evaluations of the catalytic behaviors of prepared materials under easily controlled conditions. Initial testing to establish relative product distributions and catalytic activities as a function of preparation method, pretreatment conditions, and reaction temperature has been completed for synthetic sodium- and

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potassium-graphites, a commercial iron-graphite intercalate, and, for comparison purposes, commercially available iron/alumina and cobalt/kieselguhr catalysts. Sodium- and potassium-graphite intercalates, although possessing high initial activities for the Fisher-Tropsch synthesis at 300°C, do not behave catalytically for this reaction. The Fisher-Tropsch activity of a commercially available metal aryl-reduced iron-graphite intercalate decreases with increasing reaction time but eventually becomes stabilized at a level that is relatively independent of pretreatment conditions.

**PLANS FOR THE COMING YEAR** – The catalyst testing phase of the project will be extended to investigate the Fischer-Tropsch activity/selectivity characteristics of a commercial cobalt-graphite intercalate, with particular emphasis on comparing its behavior to that of an industrial cobalt/kieselguhr catalyst. Additionally, the sensitivities of iron- and cobalt-graphites to sulfur poisoning will be explored in comparison to the corresponding behaviors of conventional supported forms of these metals.

## **ELECTRIC POWER AND THE ENVIRONMENT**

UNIVERSITY OF TEXAS, AUSTIN

DOE - \$10,228

1/1/76 - 12/31/76

Principal Investigator - E.L. Draper, Jr.

**OBJECTIVES** – “Electric Power and the Environment” was a course designed to provide high school and junior high school science teachers with a strong background in the technical, economic, environmental, and sociopolitical aspects of the generation of electric power. Advantages and disadvantages of various generation techniques were discussed with special emphasis being given to conservation, coal, and nuclear power. The cost-benefit concept was introduced as a mechanism for making rational decisions.

**RECENT WORK AND ACCOMPLISHMENTS** – Lectures were given on the topics of historical and projected energy demand; energy production techniques including oil, gas, solar, coal, lignite, geothermal, fission, fusion, refuse, hydroelectric, wind, and waves; energy conservation; waste production and environmental impact; biological effects of generation byproducts; energy transmission and distribution; and the economics of alternative electricity generation techniques. Lecturers were drawn from university, industry, and governmental agencies. Field trips and tours included the University of Texas at Austin nuclear reactor, the Texas Turbulent Tokamak fusion experiment, a combined cycle electric power plant, the Big Brown lignite mine and power plant, a uranium mine, and solar energy research projects. Special attention was given to providing the teachers with materials and sources suitable for high school classroom use. Extensive use was made of slides, film strips, and movies. Two role playing games were introduced to the participants so they could experience the variety of competing pressures on those attempting to select the type and site of power plants.

**PLANS FOR THE COMING YEAR** – The project has been completed.

## CONFERENCE ON GULF COAST LIGNITE

UNIVERSITY OF TEXAS, AUSTIN  
DOE - \$9950; Bureau of Economic Geology - \$5238  
Principal Investigators - C. Groat, W.R. Kaiser

**OBJECTIVES** – This project seeks to inform the scientific community in universities, industry, and government on the status of today's lignite developments in technology and research as they relate to the Gulf Coast area; identify and prioritize future lignite research and development needs, relative to both universities and industry; and dramatize, particularly for those in policymaking positions, the importance of lignite as an energy resource in the Gulf Coast area.

**RECENT WORK AND ACCOMPLISHMENTS** – On June 2-4, 1976, the Bureau of Economic Geology, the U.S. Energy Research and Development Administration, and Research Applied to National Needs (RANN) of the National Science Foundation held a conference on Gulf Coast Lignite: Geology, Utilization and Environmental Aspects. This conference, held in cooperation with the Center for Energy Studies (The University of Texas at Austin), drew about 500 attendees from a broad cross section of energy, environment, and geology interests. Presentations were given by representatives of public and private research groups, State and Federal government, exploration and mining companies, industrial consumers, and environmental consulting firms. A series of informal workshops focused on the geology, utilization, and environmental topics covered in the formal presentations. Two field trips were held, one to the active lignite mine and outcrops of lignite-related strata near Rockdale, Texas, and the second to the Big Brown lignite mine and large lignite-fueled steam-electric station near Fairfield, Texas.

**PLANS FOR THE COMING YEAR** – The Proceedings volume is being printed and should be available in early 1978.

## FACULTY/STUDENT PROGRAMS AT BERC, GFERC, AND LERC

ASSOCIATED WESTERN UNIVERSITIES, INC.  
DOE - \$234,000  
6/1/77 - 9/20/78  
Principal Investigator - D. Walker

**OBJECTIVES** – This program intends to provide education and training opportunities for faculty and students from primarily western universities to become acquainted with and directly involved in Fossil Fuel research problems faced by the three western Energy Research Centers and to permit qualified faculty members to assist Research Center staffs in finding solutions to fossil energy problems being explored by the Centers.

**RECENT WORK AND ACCOMPLISHMENTS** – The program originally began in June 1976 and involved three faculty members and three students during the summer months at the Laramie Energy Research Center. The participants were selected by the Center and assigned to research projects by the Center Director for the duration of the full-time appointments. During summer of 1977, faculty and students were again assigned to on-going research projects at each of the three Centers. Appointments were generally for periods ranging from 8 to 14 weeks each. Summaries of both technical and administrative aspects of each assigned project were prepared by the participants and provided to Center Directors and to the Department of Energy Headquarters Office of University Activities.

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**PLANS FOR THE COMING YEAR** — It is anticipated that the program will continue at all three Centers but in an expanded manner at both the Grand Forks and the Laramie Centers. Further, it is expected that other graduate students may be assigned projects with the research to be performed at their universities rather than at the Centers; that part-time appointments may be given to students at either one of the Centers or at their university; and that postdoctoral appointments for up to 12-month periods may be made. Occasional long-term (up to 12 months of a Sabbatical Leave) appointments may be provided to qualified faculty members for specific research programs.

**ALLOY CATALYSTS WITH MONOLITH SUPPORTS  
FOR METHANATION OF COAL-DERIVED GASES**

BRIGHAM YOUNG UNIVERSITY  
DOE - \$156,738; BYU - \$9474  
9/20/77 - 9/19/79  
Principal Investigator - C.H. Bartholomew

**OBJECTIVES** — This research program seeks to assist DOE and its contractors in evaluation of new catalyst technology for upgrading coal derived gases to synthetic natural gas. The research is focused on development of new bimetallic catalysts which are more efficient and stable than commercially available methanation catalysts and evaluation of new monolithic ceramic catalysts which may have important economic and technical advantages in the methanation process. The research objectives include careful characterization of each catalyst, a study of methanation kinetics over a range of operating conditions, investigation of catalyst deactivation by sintering, sulfur poisoning, and carbon deposition. Close communication with other researchers and interaction with other contractors to promote large scale testing and development of the best catalysts is an important aspect of the program.

**RECENT WORK AND ACCOMPLISHMENTS** — During the past year under the previous DOE contract, alumina-supported nickel and bimetallic combinations of nickel with Co, MoO<sub>3</sub>, Pt, Rh, and Ru were reactor tested to determine conversion-temperature behavior at 1 and 25 atm, effects of reactant steam and carbon deposition on catalytic activity, and differences in methane production for pelleted and monolithic catalysts having different support geometries. The conversion-temperature data show that the rate of methane production on nickel is increased 2- to 3-fold at high conversions and 4- to 5-fold at low conversions by increasing the reaction pressure from 1 to 25 atm. Addition of reactant steam dramatically increases carbon dioxide production while decreasing methane production. In carbon deposition tests at 400°-450°C (H<sub>2</sub>/CO = 2) alumina-supported Ni, Ni-Co, Ni-Pt, Ni-Rh, and Ni-Ru lose 20 to 50 percent of their activity after 7 to 10 hours, while Ni-MoO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> is completely deactivated. Monolithic-supported nickel is significantly more active for methane production than pellet-supported nickel. Thus, monolithic-supported catalysts are especially attractive for application in a high throughput recycle methanator. During the first 2 months of the new DOE contract, equipment to build two new reactor systems was ordered. Reactor testing of the same six catalysts was continued to determine the effects on methanation activity of in situ exposure to 10 ppm H<sub>2</sub>S in H<sub>2</sub> at 250°C. All of the catalysts were found to be at least partially deactivated after 12 to 36 hours exposure to H<sub>2</sub>S, while the metal surface areas subsequently measured by hydrogen adsorption fell to zero. Although approximately 10 to 15 percent of the original H<sub>2</sub> uptake was recovered after extended treatment in H<sub>2</sub> at 450°C, methanation activity was not recovered. Instead, the H<sub>2</sub> treatment resulted in a complete deactivation of every catalyst. These unexpected results enlarge understanding of the poisoning process. In addition, the data show monolithic catalysts to be no less resistant than



powders or pellets to  $H_2S$  poisoning. Technical communication and visits to other methanation laboratories are vital aspects of the research program. During the past year, the principal investigator and students participated in seven technical meetings, presented eight papers and talks, visited four methanation laboratories, and received nine visitors in connection with this research program. The BYU laboratory is presently in active communication with over 20 other methanation laboratories in the United States and Europe. Training of students is another important part of this program.

**PLANS FOR THE COMING YEAR** — Construction of two new recycle reactor systems (one quartz and one stainless steel) will continue during the next 1 to 2 months. When completed, the quartz reactor will be used to investigate methanation kinetics of nickel, ruthenium, and nickel bimetallic catalysts in the absence and presence of dilute  $H_2S$  at low pressures. The stainless steel Berty reactor will be used to study methanation kinetics at high pressure (up to 75 atm). The thermal stability and resistance to carbon deposition of the same catalysts will also be investigated. Physical and chemical properties of each catalyst will be characterized by means of chemisorption, Auger, ESCA, X-ray, and electron microscopy measurements. Closer interaction with industrial and government researchers to achieve large-scale testing of the best catalysts is also planned.

### MIXING AND GASIFICATION OF COAL IN ENTRAINED FLOW SYSTEMS

BRIGHAM YOUNG UNIVERSITY  
DOE - \$244,378; BYU - \$12,866  
5/1/77 - 4/30/79  
Principal Investigator - L.D. Smoot

**OBJECTIVES** — This research program seeks to develop an understanding of physical and chemical rate processes that occur during gasification of entrained, pulverized coal. The effect of reactor geometry is being emphasized in a series of entrained flow, laboratory-scale experiments. Mathematical computer design models for coal gasifiers and combustors are being developed and evaluated.

**RECENT WORK AND ACCOMPLISHMENTS** — Over 130 cold-flow jet mixing tests simulating coal gasifier operating conditions have been conducted using an existing cold-flow test facility. Effects of injection angle, mixing chamber size, flow velocity and temperature, and coal size and loading have been measured. The design and assembly of the laboratory coal gasifier facility has been completed and gas flames ignited and stabilized on several occasions. The first coal flames have also been stabilized. Alteration of the coal feeder has been completed to obtain uniform coal feed rates. Design and fabrication of a prototype of the sample train for removing gas-coal samples from inside the gasifier have also been completed and are being tested. Iron in the coal ash is being considered as a better key component tracer for the coal than the total ash which was shown to be partially volatile at higher temperatures and partially soluble in the probe coolant water. The development of the one-dimensional gasifier computer code has been completed, and work has been initiated (1) to explore effects of model parameters and test variables on predicted flame structure using the code and (2) to apply the one-dimensional computer code to entrained gasifiers. Two industrial entrained gasifier units are presently being examined with this code. Generalized two-dimensional model development is continuing, emphasizing integration of model components. The technical basis of the model has been improved by accounting for micromixing processes. Sample computations have been completed for the laboratory gasifier.

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**PLANS FOR THE COMING YEAR** – The operational checkout and shakedown of the laboratory-scale gasifier will be completed and parametric studies will be made investigating the effects of operational conditions, geometry, and coal type. Additional cold-flow mixing tests will be completed to investigate the mixing characteristics of nonparallel secondary injection combined with an enlarged recirculation mixing chamber. The completed one-dimensional gasifier model will be used to make parametric studies to investigate the effects of controllable parameters on the gasification process. The general multi-dimensional model integration will be completed and debugged, and comparison with experimental test results will be initiated.

## **SIALON REFRACTORIES FROM CLAY AND COAL**

UNIVERSITY OF UTAH  
DOE - \$120,000  
6/1/76 - 5/31/78  
Principal Investigator - I.B. Cutler

**OBJECTIVES** – Sialon, a solid solution of silicon nitride and aluminum oxynitride, can be obtained by reaction of clay and coal with nitrogen. The properties of sialon are comparable and in some instances superior to silicon carbide and to silicon nitride. The objective of this research will be to develop a practical scheme for producing sialon brick for test in coal gasification facilities. The project will determine the essential process parameters (e.g., temperature, nitrogen partial pressure, and carbon-to-clay ratio) for production of sialon from clay, coal, and nitrogen. Tolerance for gaseous and chemical impurities will also be detailed. Both grain and refractory brick will be produced on a laboratory scale and their properties examined.

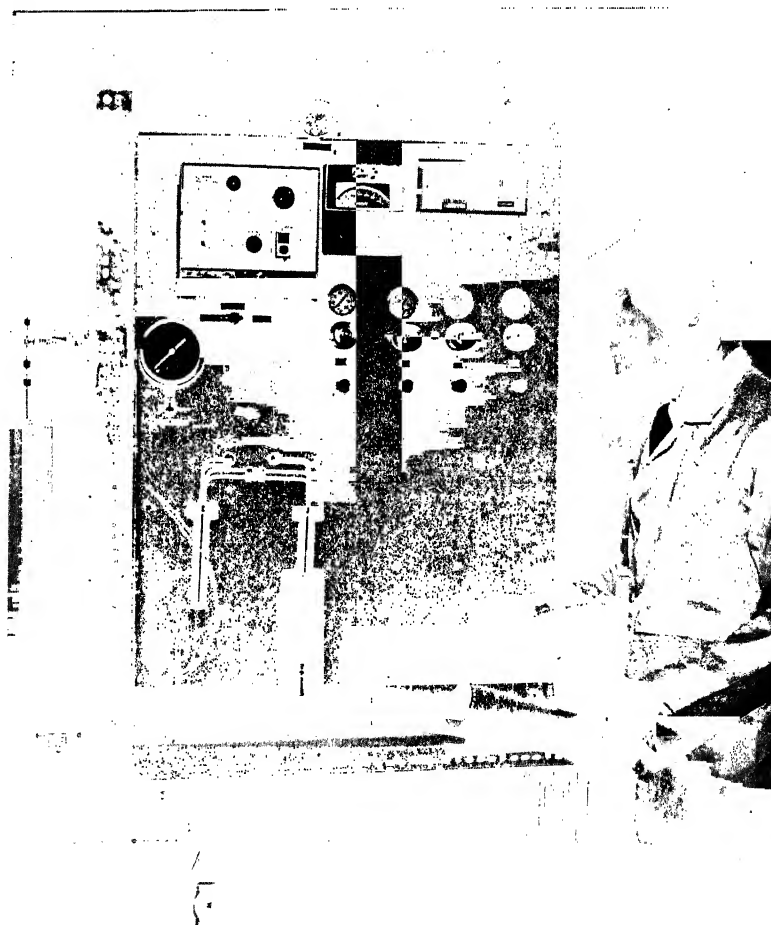
**RECENT WORK AND ACCOMPLISHMENTS** – Production of sialon refractories from clay is a two-step process requiring reaction of clay, carbon (coal), and nitrogen (perhaps from air) to produce a suitable powder at temperatures below 1440°C; and sintering of a sialon powder compact at about 1650°C. To date it is known that the clay-to-carbon ratio is important to the production of a single phase sinterable powder, the temperature cannot be raised above about 1440°C without yielding SiC in preference to sialon, and iron acts as a catalyst in the formation kinetics of sialon. Appreciable amounts of hydrogen can be present both during the reaction as well as during sintering without altering the processes. Iron in the sinterable sialon powder can be largely removed by magnetic separation. More than adequate strength can be obtained from sintered sialon for use as a refractory material. Preliminary work shows that at least 30,000 psi can be obtained from bend tests on sintered sialon. Since the coefficient of thermal expansion is also low (in the 25° to 1000°C range), this material should have good heat shock resistance. A continuous reaction system has been developed to produce sinterable sialon powder at a rate of approximately 1 kg/d. This powder is sintered into refractory grain for use in the production of brick. Process parameters have been determined for these steps.

**PLANS FOR THE COMING YEAR** – Production of refractory brick and determination of properties of the final products of sintering will be accomplished. The effect of impurities contained in the clay will be investigated.

## PROCESSES FOR LIQUEFACTION AND GASIFICATION OF WESTERN COALS

UNIVERSITY OF UTAH  
DOE - \$2,576,880; U of Utah - \$235,120  
6/1/75 - 5/31/79  
Principal Investigator - W.H. Wiser

**OBJECTIVES** – The 16 different research projects conducted in connection with this contract cover evaluation of process concepts for liquefaction and gasification of coal; studies of the fundamentals of catalysis as applied to coal liquefaction and gasification; studies of the mechanisms involved in coal liquefaction and gasification; and determination of the properties of coal and of coal conversion products. The information developed in these studies will assist in optimization of coal liquefaction and gasification processes, will assist materially in the application of coal to the solution of energy problems now facing the United States and other nations of the world, and will provide parts of the basic training for students, particularly at the graduate level, who will eventually assume responsible positions in the industries associated with energy production, conversion, and utilization.



• Micro-Catalytic Flow Reactor

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**RECENT WORK AND ACCOMPLISHMENTS** – Highlights from the broad range of projects have to do with comparison of the chemical structure of a coal hydrogenation liquid with the structure of solvent refined coals and synthoil samples; comparison of hydropyrolysis and thermal cracking of coal-related model compounds; measurement of the reaction kinetics of the CO/H<sub>2</sub> reaction over CoCu/Al<sub>2</sub>O<sub>3</sub> catalyst; and measurement of the hydrogen exchange properties of Lewis and Bronsted acids to verify that ZnCl<sub>2</sub>/SiO<sub>2</sub> behaves as a Bronsted acid. An infrared spectrometric study of solvent refined coal products from a tetralin-coal dissolution system has been completed. Ultraviolet spectrometric analysis of the coal extract product shows spectral changes with increased reaction times: short reaction time products show spectra similar to vitrain or hydroaromatic structures, and spectra of longer reaction period products show naphthalene-like patterns. This may indicate the development of an average two-ring structure in the liquid product under extended reaction conditions. Conclusions from the comparison of thermal cracking and hydropyrolysis of n-hexadecane are as follows: (1) Paraffin/olefin ratios in the C<sub>2</sub>-C<sub>4</sub> fractions are much higher in hydropyrolysis than in thermal cracking and increase sharply with an increase in hydrogen pressure in the range of 500 to 2000 psig; (2) the relative yields of CH<sub>4</sub> are considerably lower while those of C<sub>3</sub>-C<sub>5</sub> components are markedly higher in hydropyrolysis than the yields observed for these components in thermal cracking; for components >C<sub>5</sub>, only olefins formed in thermal cracking, whereas mostly paraffins formed in hydropyrolysis; no aromatic derivatives or products higher than the starting material are formed by hydropyrolysis, whereas in thermal cracking, especially under pressure, there is considerable formation of aromatics and high-boiling products; and (5) differences in product distribution, in particular for C<sub>1</sub>-C<sub>5</sub> components, can be attributed to anticipated differences in the reaction mechanism of the two processes.

A process variable study was carried out on one of the most promising CoCu/Al<sub>2</sub>O<sub>3</sub> catalysts to establish the basic kinetics of the reaction. The reaction rate was found to be first order in H<sub>2</sub> and inverse square-root order in CO, with an activation energy of 25 kcal/mole. No promoting effect of K or Na was observed. Catalyst characterization studies made on the CoCu/Al<sub>2</sub>O<sub>3</sub> catalyst system revealed a synergistic relationship in that metal dispersions for the composite catalyst were appreciably higher than for the single-component catalysts; cobalt helped to better disperse the copper, while copper aided in reduction of the cobalt to the active state. An alloy or metal cluster is believed present on the activated catalyst. Catalyst activities correlated well with total metal area as measured by oxygen chemisorption. The deuterium exchange reactions of various hydrocarbons over catalysts of known acid type were compared to exchange reactions for ZnCl<sub>2</sub>/α-Al<sub>2</sub>O<sub>3</sub> and ZnCl<sub>2</sub>/coal. The ZnCl<sub>2</sub> impregnated samples demonstrated exchange properties identical to those for a Bronsted acid. These results suggest that the Bronsted acid character of the ZnCl<sub>2</sub> · H<sub>2</sub>O species may play an important part in hydrogen transfer reactions in coal.

**PLANS FOR THE COMING YEAR** – Each of the various specific projects which are part of this total contract will be pursued theoretically and experimentally. Publications, theses, and presentations will be made as deemed appropriate.

## GAS-SOLIDS INTERACTIONS IN FLUIDIZED-BED DISTRIBUTION ZONE

UNIVERSITY OF VIRGINIA  
DOE - \$116,947; UVa - \$3055  
6/15/76 - 7/31/78  
Principal Investigator - D.J. Kirwan

**OBJECTIVES** – This program seeks to investigate the interactions between gases and solids in the distribution zone of a fluidized bed with particular emphasis on delineation of the regimes of stagnant (defluidized), moving but nonfluidized, and fluidized solids. Improper design of the distribution system for gases entering a fluidized bed can have a number of deleterious effects. Successful correlation of solids distributions patterns with distributor geometry and operating variables such as bed superficial velocity, gas entry velocity, and entry port spacings will lead to improved distributor design methods for large-scale fluidized-bed installations involving coal.

**RECENT WORK AND ACCOMPLISHMENTS** – The use of a miniature self-heating thermistor probe to identify the type of gas-solids behavior in the distribution zone has been extended to the three-dimensional probing of five single air entry reactors ranging from 3.4 to 8.75 in. in diameter with a fluidized solid of 60 $\mu$  petroleum cracking catalyst. The results of these probings have been used to plot three-dimensional scans of the several gas-solids regime interfaces in the reactors tested at two superficial velocities and at three air entry velocities. A larger air handling system was placed in service and used to successfully probe single entry air reactors with 6 to 16 mesh crushed limestone at a superficial velocity of 10 ft/sec. Two reactors of 4.5 and 5.5 in., respectively, were used to test two air entry velocities. A modified self-heat thermistor probe with increased mechanical strength was developed for use in the limestone testing program. A seven port reactor 19 in. in diameter with 60 $\mu$  petroleum catalyst is being probed to determine the interactions and effects of adjacent air entry points on the several gas-solids regime interfaces in the distribution zone.

**PLANS FOR THE COMING YEAR** – Probing of the 19-in. seven-port reactor will be completed, and a 14-in., seven port reaction will be installed and probed to study the entry port interactions at a higher superficial velocity. The results of these probings will be analyzed and used to plot three-dimensional interfaces. A model to relate the several parameters involved will be developed and used to predict distributor changes to reduce the magnitude of poorly fluidized regimes. A modified distributor arrangement based on this prediction will then be installed and tested.

## STABILIZATION OF ROTATING MACHINERY FOR FOSSIL FUEL

UNIVERSITY OF VIRGINIA  
DOE - \$319,667; UVa - \$41,375  
8/15/76 - 8/14/79  
Principal Investigator - D.W. Lewis

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**RECENT WORK AND ACCOMPLISHMENTS** – The experimental facilities have been built, instrumentation has been acquired, and several forms of precision bearings with two rotors for a specially designed experimental rotor rig have been delivered to UVa. Papers on several phases of the theoretical work have been completed. A computer program has been completed which provides essential data of modeled rotor systems and calculates the modal weights, modal damping, and amplification factor for the critical speeds of a rotor.

**PLANS FOR THE COMING YEAR** – The experimental facilities and special bearings that have been delivered to UVa will be tested with the goal of bringing theory and actual usability closer together. Actual data received on real machines, including the Space Shuttle main engine, will be analyzed and computer modeling pursued so that theory and machine data can be understood. Work will be directed toward multimass rotors for calculating the stability, the transient response, and fluid film bearing characteristics.

### **STIRRED-TANK REACTOR EXTRACTIVE COAL HYDROGENATION PROCESS**

VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY

DOE - \$191,996; VPI and SU - \$24,657

6/27/74 - 10/1/78

Principal Investigator - G.H. Beyer

**OBJECTIVES** – This research intends to economically convert coal to a clean solid and/or liquid fuel by developing and applying chemical engineering techniques to improve process flowsheets by using a continuous stirred-tank reactor with residence times long enough to open the coal for sulfur removal, yet short enough to minimize hydrogen consumption.

**RECENT WORK AND ACCOMPLISHMENTS** – A continuous loop reactor system has been built and operated. The loop reactor uses a recycle pump for rapid recirculation of coal slurry through a “loop” made up of the pump and a tubing coil immersed in a hot fluidized bed. Residence time is controlled by the rate at which new coal slurry is pumped to and overflowed from the loop. Residence times of less than 7 minutes at 425°C have been obtained. The loop reactor concept accomplishes rapid heating with excellent heat and mass transfer. Relatively short residence times permit study of the early decomposition products of coal, so-called “prompt coal,” that may represent a uniquely reactive material. Using a loop reactor, the sulfur content of a 4.25 percent Illinois coal has been reduced to 3.4 percent using only the hydrogen indigenous to the coal itself.

**PLANS FOR THE COMING YEAR** – The loop reactor system will be operated at various temperatures, pressures, and gas environments to explore the unique properties of “prompt coal.”

### **CO DISINTEGRATION OF REFRACTORIES IN COAL GASIFIERS**

VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY

DOE - \$132,559

6/1/77 - 5/30/79

Principal Investigator - J.J. Brown, Jr.

**OBJECTIVES** – This investigation seeks to determine whether or not CO disintegration is likely to occur in the refractories used in coal gasifiers and to define the conditions under which this can be expected to become a serious problem.

**RECENT WORK AND ACCOMPLISHMENTS** – Refractor castables of the 90<sup>+</sup> percent Al<sub>2</sub>O<sub>3</sub> (calcium aluminate cement plus tabular and calcined alumina) and 50<sup>+</sup> percent Al<sub>2</sub>O<sub>3</sub> (calcium aluminate cement plus calcined kaolin) when exposed to CO at 500°C and atmospheric pressure for 100 hrs were found to undergo no serious deterioration. When these castables were doped with 0.5 or higher weight percent metallic iron, extensive disintegration occurred after 100 hrs. The 90<sup>+</sup> percent castable appeared to be more resistant to disintegration than the 50<sup>+</sup> percent castable. Similar additions of up to 2 weight percent Fe<sub>2</sub>O<sub>3</sub> (hematite) did not lead to any visible cracking in either castable; however, a reduction in the strength of both castables was noted. A third refractory type, 90<sup>+</sup> percent alumina phosphate bonded ramming mix is being examined at the present time.

**PLANS FOR THE COMING YEAR** – Because it has been demonstrated that CO disintegration is a potential refractories problem in coal gasifier plants, it is important to determine the conditions under which this will occur. This investigation will therefore study the effects of gas mixtures, pressure, temperature, impurities in the refractories (iron compounds), impurities in the coal ash (most notably alkali compounds), and type of refractory on the resistance to CO disintegration.

## DESIGN OPTIMIZATION IN UNDERGROUND COAL SYSTEMS

VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY

DOE - \$456,000; VPI and SU - \$81,000

12/10/73 - 6/10/78

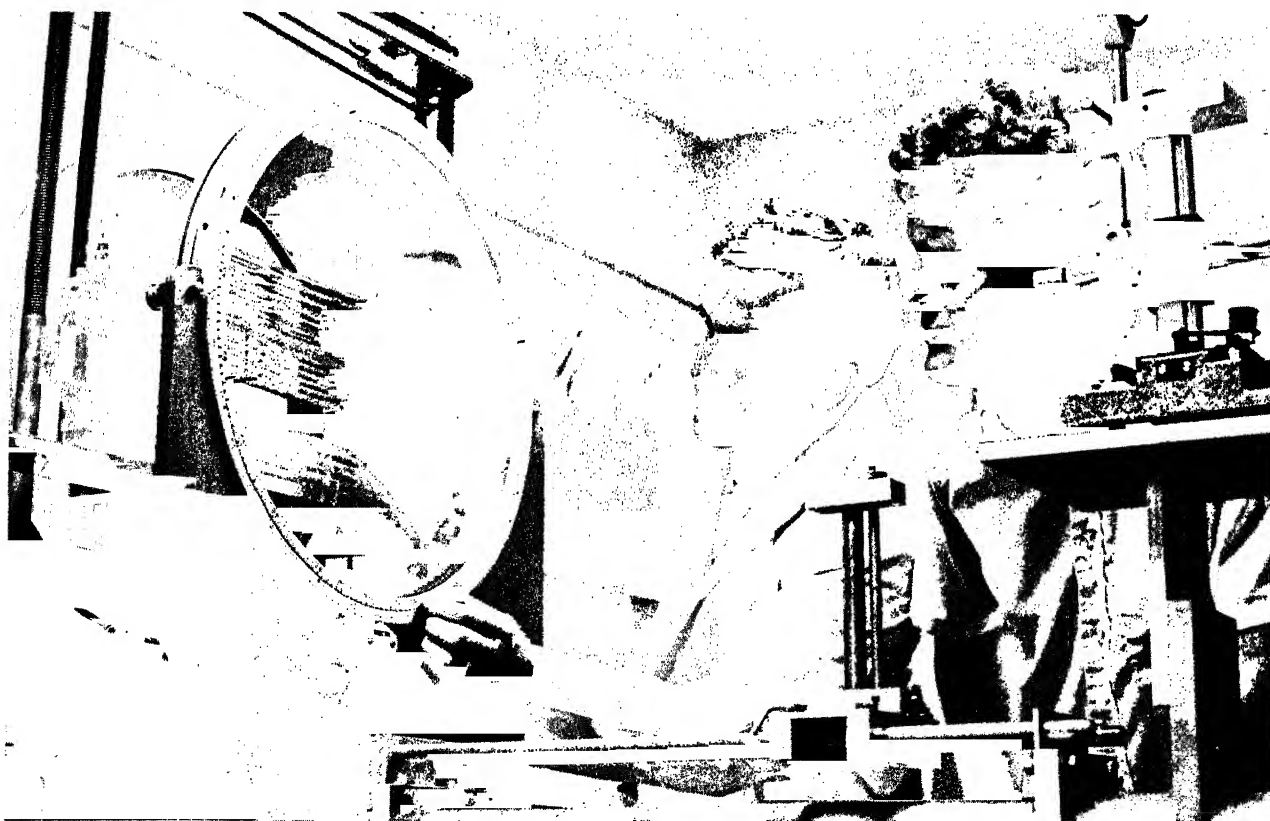
Principal Investigators - J.R. Lucas, C. Haycocks

**OBJECTIVES** – This program aims at facilitating long-range planning of underground coal systems. In regard to structural characteristics of coal, it was necessary for design purposes to develop a strength relationship between cylindrical and cubical test specimens and to determine the effects of loading rate and moisture content in order to formulate a test procedure for the field engineer. A longwall mining simulator is being developed from the data obtained from exploration boreholes to model the mining environment, geologic environment, and strata behavior. This will permit a more precise design for specific equipment selection and application. A primary objective of the research is to establish the design criteria for roof trusses for a broad range of domestic mining conditions. The systems optimization effort is aimed at the maintenance and improvement in output and input formats used in existing program simulation for mine-design programs previously developed at this institution, and the development of programs to optimize LHD applications and analyze innovative mining systems.

**RECENT WORK AND ACCOMPLISHMENTS** – As a result of testing cubical and cylindrical specimens from a wide variety of U.S. coal seams, an empirical relationship was developed relating the strength of samples to the two different geometries. Size versus strength relationships as well as the effect of loading rates and moisture content were also investigated. Standardized field testing



*Digital Programmable Control Unit for Large Coal-Specimen Test on  $10^6$  MTS Testing System*



*18-Inch Diffused-Light Polariscope Used for Analyzing Model Representing a Roof Truss in Mine-Roof Strata*



systems. Input data are primarily derived and extracted from exploration boreholes. The roof truss has been evaluated using a body-loaded photoelastic model in the laboratory. For midspan-type failures, it was found that an inclination of  $45^{\circ}$  appears to be most effective for increased support, and effectiveness of the truss system increases with decreasing truss span. For ribside shear failures, inclinations of between  $45^{\circ}$  and  $60^{\circ}$  proved to be most effective. For shorter-span trusses, the hole depth should be sufficient to place the anchor beyond the rib face. Field studies have established fairly conclusively that there is no friction loss across blocking points of trusses with routine inclinations (approximately  $45^{\circ}$ ), and initial truss loading apparently does become relieved with time. Existing systems optimization programs have been upgraded and maintained and a new LHD simulator is in the process of being completed.

**PLANS FOR THE COMING YEAR** — The completion of all facets of the scope of work will be achieved.

### **SUPPORTED MOLTEN ELECTROLYTE CATALYSTS**

VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY

NSF - \$187,300

4/1/73 - 1/31/77

Principal Investigator - P.R. Rony

**OBJECTIVES** — This project intends to construct an efficient catalyst screening microreactor and to search for new or improved catalyst compositions of matter for fuel-to-fuel conversion reactions such as the water-gas shift reaction, catalytic methanation, and related reactions. Attention was focused upon hybrid catalysts called supported molten electrolyte catalysts and supported liquid-phase catalysts which contain a catalyst solution dispersed within a porous refractory metal oxide support such as alumina or silica.

**RECENT WORK AND ACCOMPLISHMENTS** — An 8080A-based microcomputer was used to automate the operation of a specially built catalytic microreactor that contained seven valves and three temperature controllers from VALCO Instruments, Inc. The interfacing of the reactor to the microcomputer was achieved using TTL integrated circuit chips. Hardware circuits were developed to sequence four multiport and two bypass pneumatic valves; control the operation of a high-pressure sampling valve for a gas chromatographic column; output digital setpoints to three different temperature controllers; detect whether or not the reactor system reached the required setpoint temperature; and input chromatographic data obtained from a digital integrator. A 2-kilobyte operating system was developed to coordinate the operation of the driver routines which operated individual items of mechanical equipment and permitted an operator who was not familiar with assembly language to operate the system directly from a CRT display. The operating system was structured around a polling loop that handled all input/output operations, including the timing of different events. Fourteen commands used by the operator were incorporated into the software via a key word list structure command table. Several experimental runs demonstrated the reproducibility of the sampling valve and the operation of the entire reactor system for the methanation reaction using a ruthenium-based catalyst. The total system permits the sequential study of seven different catalysts for eight different feed streams of eight different valve-selected space velocities, and of a variety of temperatures and pressures.

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**PLANS FOR THE COMING YEAR** – The project ended on October 31, 1976, but a time extension was obtained at no additional cost to permit the M.S. Thesis to be written and submitted. Work on the screening of selected catalysts is planned.

## CHEMISTRY OF DONOR SOLVENT COAL LIQUEFACTION

VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY

DOE - \$308,090; VPI and SU - \$183,020

9/1/77 - 8/31/79

Principal Investigators - A.M. Squires, J.G. Dillard, H.C. Dorn, P.R. Rony, L.T. Taylor

**OBJECTIVES** – This program will develop and apply analytical techniques to study the chemistry of donor solvent liquefaction of coal. Reaction mechanisms and reaction kinetics will be investigated as the analytical techniques become ready, with special emphasis upon regressive reactions that sometimes convert significant portions of the coaly matter to mesophase and semi-coke. An understanding of the regressive reactions may lead to improvements in coal liquefaction processes from two standpoints: minimizing trouble that sometimes arises from deposits of semi-coke in a donor solvent coal liquefaction reactor; and minimizing the rate of deactivation of catalytic surfaces, if they are present in the reactor, through formation of deposits upon such surfaces.

**RECENT WORK AND ACCOMPLISHMENTS** – A batch-stirred microautoclave is being constructed to prepare samples of reacted coal for chemical analysis. Five samples of solvent refined coal (SRC) solid have been obtained from the pilot plant at Wilsonville, Alabama (produced from Pittsburgh No. 8 coal, Western Kentucky No. 9 and 14, Illinois No. 6, a high-sulfur Illinois coal, Monterey, and a Western coal, Amax). Separation of the portion of the SRC solids that is soluble in tetrahydrofuran (THF) has been accomplished according to effective molecular size via high performance gel permeation chromatography with a number of candidate column packing materials, and Bio-Beads SX-4 (4 percent cross-linking) has been identified as a preferred material. Separated fractions of the SRC solids have been examined by pulsed Fourier transform  $^1\text{H}$  and  $^{13}\text{C}$  nuclear magnetic resonance (NMR) spectroscopy. Paramagnetic relaxation reagents as well as gated  $^1\text{H}$  decoupling sequences have been used to suppress undesirable nuclear Overhauser effects and long spin-lattice relaxation times, and thereby to secure quantitative  $^{13}\text{C}$  NMR measurements. Mixtures of coal liquid samples and a reference species, hexamethyldisiloxane, have been studied.

**PLANS FOR THE COMING YEAR** – Coal liquefaction runs will be made in the microautoclave both with good donor solvents, such as tetralin, and also with poor solvents, such as naphthalene, over reaction times ranging from about 1 min to about 1 hr. Chromatographic separation studies will be extended to columns of radically different design, separations of the chloroform- and benzene-soluble portions of SRC solids, affinity-type separations of sized fractions, separation of acid-base-neutral fractions via gel permeation chromatography, and model compound studies with Bio-Beads in different solvents in order to establish exclusion limits, elution volume-size relationships, etc. Quantitative  $^1\text{H}$  and  $^{13}\text{C}$  NMR studies will continue to be used for chemical characterization parameters. Operating conditions will be studied for determination of certain oxygen, nitrogen, and sulfur functional groups in chromatographic fractions of liquefied coal by a procedure involving trifluoroacetylating selected functionalities in a given fraction and identifying the functionalities and determining them quantitatively by  $^{19}\text{F}$  NMR spectroscopy.

## FATE OF FUEL-SULFUR IN BACKMIXED CONTINUOUS COMBUSTION

WASHINGTON STATE UNIVERSITY

DOE - \$74,816; WSU - \$3741

2/1/76 - 7/31/78

Principal Investigator - P.C. Malte, W.L. Grosshandler

**OBJECTIVES** – Laboratory experiments are conducted to provide a data base on sulfur compounds that form in utility and industrial combustors that burn pulverized coal. Such combustors employ backmixing to stabilize the flame. The current research is designed to overcome deficiencies in available data in this program area. Its payoff will be a better understanding of sulfur species in combustion systems so that utility and industrial boilers can be designed to minimize the deleterious effects of sulfur on system corrosion and on pollutant emissions. The specific program objectives are the kinetics of  $\text{SO}_3$  formation; systematic measurement of the sulfurous products of fuel-rich combustion (i.e.,  $\text{H}_2\text{S}$ ,  $\text{COS}$ ,  $\text{CS}_2$ , mercaptans, and polycyclic hydrocarbons); and measurements of the composition and kinetics of the volatilization of sulfur from pulverized coal and from model sulfur compounds (i.e., thiophene).

**RECENT WORK AND ACCOMPLISHMENTS** – The experimental facilities have been completed except for the final design and fabrication of the gas sampling systems for pulverized coal combustion. For this case, two sample probes are being constructed: one for gas, and one for particulate matter. The pulverized coal burner has been tested, demonstrating that pulverized coal can be burned in a high-intensity jet-stirred, primary zone of the combustor. The pulverized coal burner is designed to stabilize the jet-stirred primary zone on propane-air, and then to add the pulverized coal as the sulfur additive. In this way, the gaseous environment into which the coal is injected is approximately known. Measurements are also scheduled with the burner stabilized completely on pulverized coal. Production measurements are underway in the jet-stirred Longwell reactor for the sulfurous products of propane-air combustion doped with thiophene ( $\text{C}_4\text{H}_4\text{S}$ ),  $\text{H}_2\text{S}$ , and  $\text{SO}_2$ . These measurements also show that the decomposition of thiophene is incomplete when the fuel-air mixture is above 40 percent excess fuel, and the decomposition is markedly temperature sensitive. Work on the analysis of gas-sample probes has been completed, showing that there is little practical distortion of the  $\text{SO}_2$  concentration, but the  $\text{SO}_3$  concentration will be distorted to lower levels in the probe unless the probe pressure is low ( $>0.05$  atm) or the probe is completely cooled. This area of research has also involved the passage of  $\text{COS}$ ,  $\text{H}_2\text{S}$ , and  $\text{SO}_2$ , in either air or  $\text{N}_2$ , through hot tubes of stainless steel (to  $1250^\circ\text{K}$ ) and quartz (to  $1700^\circ\text{K}$ ). The optical absorption measurement of background concentrations of OH-radicals and O-atoms in the jet-stirred reactor has been completed. Currently, testing of a new dual beam optical system is underway that may afford sufficient sensitivity to observe some of the above sulfur species in absorption.

**PLANS FOR THE COMING YEAR** – The experimental matrix on the sulfur species that occur in the gas-phase Longwell reactor will be completed, and the initial measurements of sulfur fate in the pulverized coal burner will be made. Measurements regarding the  $\text{SO}_3/\text{SO}_x$  ratio will begin, and the optical system will continue to be upgraded. It is also believed that systematic laboratory measurements of sulfur-nitrogen interaction and sulfur-alkali interaction during pulverized coal combustion are needed, and suitable proposals are planned.

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## HIGH-PRESSURE ROTARY PISTON COAL FEEDER DESIGN

WEST VIRGINIA UNIVERSITY  
DOE - \$102,613; WVU - \$2329  
10/74 - 12/78  
Principal Investigator - H.T. Gencsoy

**OBJECTIVES** — This program intends to design a dry coal feeder with discharge capabilities of 1500 psig and 350°F. The critical design problems are the rotary-piston mechanism, lubrication, wear, and sealing of feeder components. The initial design in Phase I is for 100 psig discharge pressure at 350°F with a feed capacity of 200 to 1000 lbs/hr. Design problems associated with the rotary feeder must be solved at these modest conditions before higher pressures and flows can be achieved. Phase II consists of fabrication and testing of the prototype pump. Phase III will extend the design to 1500 psig with feed rates of several tons of coal/hr.

**RECENT WORK AND ACCOMPLISHMENTS** — In Phase I, a feeder with 100 psig discharge capability was designed, and all related problems such as lubrication, wear, and seals were investigated. Currently in Phase II, a prototype feeder is being built for seal testing only. Special wear-compensating type seals were developed for this application. Arrangements are presently being made for their fabrication.

**PLANS FOR THE COMING YEAR** — Fabrication of the feeder components will be completed, and the prototype test unit will be assembled. Feasibility and development capabilities of the new seals will be evaluated first under operating pressure conditions only. Seal wear and gas leakage rates will be investigated, and if necessary, various other design concepts will be investigated for successful seal operation. Following the seal testing, a complete prototype feeder will be built and tested for general performance of its various components such as bearings, cams, cam followers, linear ball bushings, etc. Depending on the potential for success in Phase II, this work will be extended in Phase III for the design of a coal feeder to deal with higher pressures and feed rates.

## ASH REMOVAL FROM COAL-DERIVED LIQUIDS

WEST VIRGINIA UNIVERSITY  
DOE - \$202,627  
4/76 - 3/78  
Principal Investigator - J.D. Henry, Jr.

**OBJECTIVES** — A process will be investigated which involves the extraction of hydrophobic coated mineral matter from a coal-derived liquid to an aqueous phase or collection of the mineral matter at the interface between the phases. Another objective of this research is to develop an alternative to vary expensive conventional processes such as filtration and centrifugation to remove mineral matter from coal derived liquids.

**RECENT WORK AND ACCOMPLISHMENTS** — Most of the research during the last year has been directed toward an understanding of the wetting characteristics of the asphaltic coated particles. Further experiments with the asphaltic coated mineral matter in model solvents such as xylene confirm the partial detergency mechanism. This model explains the influence of the distribution coefficient mixing time dependence, and the influence of surfactant concentration, water/oil ratio, and mixing speed at long contact times. A free energy analysis of the particle distribution process has been completed. The model indicates that the three-phase contact angle and the ratio of the

water droplet to mineral matter particle size will have a critical effect on the feasibility of the particle distribution process. An experimental technique has been developed to measure the three-phase contact angle on discs which are formed from the asphaltic coated mineral matter recovered from coal-derived liquids. Modified basic sediments and water tests have been developed to observe particle removal to the dispersed phase in a coal derived liquid. The same surfactants that lower the three-phase contact angle on the mineral matter discs also give significant mineral matter recovery from solvent refined coal filter feed. Particle removal as high as 60 to 80 percent in a single stage of contact was observed. Also, it was observed that surfactants can be ranked on their effectiveness to remove particles from coal derived liquids based on their three phase contact angle. Electrical coalescence experiments are underway to identify the mechanisms of electrical coalescence. Experimental equipment which will permit measurement of the coalescence rate by photographic techniques has been constructed and is being tested.

### COAL MINERALS BIBLIOGRAPHY

WEST VIRGINIA UNIVERSITY  
DOE - \$83,200  
6/15/77 - 6/14/78  
Principal Investigator - J.W. Leonard

**OBJECTIVES** – This project intends to conduct a comprehensive literature survey to collect information on minerals occurring in coal and in coal by-products resulting from the handling and processing of coal. The literature will include information on coal minerals as they occur naturally within the seam, as they are present following extraction and preparation, and as they affect and are affected by coal gasification, combustion, and liquefaction processes.

**RECENT WORK AND ACCOMPLISHMENTS** – Initial work on this project involved investigation of various computer information retrieval services. After consideration of the available electronic retrieval services, the following four were selected: GEOREF, CHEMCON, Engineering Index, and RECON. It was necessary to supplement the computer searches with an extensive manual search of the available literature. The two largest data bases which have been covered manually are Chemical Abstracts and Engineering Index. Additional abstracting services such as the DOE Energy Research Abstracts, the Index of Geology, and the list of publications from various state geologic surveys have also been examined. All of these sources have yielded a total of approximately 2000 potential references.

**PLANS FOR THE COMING YEAR** – A detailed study of those references identified as potentially useful for annotation will be made to determine which are to be included in the final report. Each report that fits the criteria of the project will then be annotated, indexed, and published in the format of a final report. Because of the large number of references found during the literature search, a substantial man-hour commitment is anticipated both in reviewing articles and in preparing the final document, *Coal Minerals Bibliography*.

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## PREDICTION MODEL FOR COMPOSITIONAL CHANGES WITHIN A COAL

WEST VIRGINIA UNIVERSITY

DOE - \$186,000

6/76 - Continuing

Principal Investigators - J.J. Renton, A.C. Donaldson

**OBJECTIVES** – This study applies basic sedimentological and geochemical principles to coal and thereby demonstrates that the compositional variability within a body of coal, as within any other sedimentary rock, reflects the basic depositional and early diagenetic history. The compositional variability is also systematic in distribution and allows the formulation of a predictive sedimentological-geochemical model which in turn can be applied to the evaluation of a specific coal, on either a local or a regional basis, to a specific conversion process based upon the known compositional requirements of that process. This will allow a more accurate, intelligent evaluation of the coal reserves of this country to be made relative to the conversion process to which the coal is best suited and will thereby ensure the maximum utilization of the energy represented by those remaining coal reserves.

Such a model will also allow specific conversion characteristics of a coal to be evaluated before mining; for example, the evaluation of the inorganic composition of the coal as it relates to problems of conversion efficiencies, to boiler fouling or to the poisoning of catalytic conversion processes. In fact, any coal utilization process that is in any way dependent upon the composition of coal will have direct use of information made available by a predictive compositional coal model.

**RECENT WORK AND ACCOMPLISHMENTS** – A within-a-mine study involving a three dimensional investigation of the inorganic composition within a surface mine of the Waynesburg coal near Morgantown, West Virginia, has been completed. The study consisted of a detailed compositional investigation of 347 6-inch increment samples systematically removed from the coal at 23 sampling locations. Maps and cross sections have been prepared for most of the basic compositional parameters. The map and cross sections show definite patterns which in most cases can be related to depositional and early diagenetic history of the coal. A smaller study of 209 lithotype samples taken stratigraphically at three sampling sites has been petrographically analyzed. Statistical tests are being run between the organic and inorganic parameters. The study so far has shown positive correlations between percent vitrinite in the coal and the percent kaolinite of the low temperature ash and between percent vitrinite and percent organic sulfur. This study also includes a detailed study of the iron disulfide (pyrite and marcasite) distribution within the coal based upon grain morphology as well as organic and inorganic associations.

The study has been extended to a more regional scale where the coal and its enclosing stratigraphy are being investigated over an area of about 500 square miles. Maps of coal thickness, partings, and overlying as well as underlying strata have been generated in order to formulate a more general regional model. Definite correlations have been made between what has been observed in the Waynesburg coal and recent observations in modern coal forming swamps. These data definitely indicate that the paleo-depositional and early diagenetic imprint exists within the coal and can be recognized. The importance of this fact is that it will allow the formulation of a regional predictive compositional model for coal.

## COAL THERMOLYSIS—A FUNDAMENTAL CHEMICAL KINETICS APPROACH

WEST VIRGINIA UNIVERSITY  
DOE - \$40,000; WVU - \$2784  
9/1/77 - 8/30/79  
Principal Investigator - S.E. Stein

**OBJECTIVES** – The objective of this research is to demonstrate the feasibility of analyzing coal conversion chemistry from a basic chemical kinetics standpoint. The most immediate goal is to develop reliable methods for modeling liquid-phase pyrolyses of aromatic compounds and mixtures. Specific objectives are to perform thorough analyses of products from pyrolysis of different classes of aromatic substances characteristic of coal and coal liquids and to develop kinetic models based on elementary rate parameters.

**RECENT WORK AND ACCOMPLISHMENTS** – The experimental set-up for liquid-phase pyrolysis is under construction and optimal analytical schemes are being tested.

**PLANS FOR THE COMING YEAR** – Pyrolyses of selected aromatic and polyaromatic hydrocarbons will be performed over as wide a range of temperatures and reaction times as feasible. Compositions of product mixtures determined by gas chromatography and mass spectrometry will be compared with predictions of initial kinetic models. The origin of any differences between experimental results and predictions will be investigated carefully.

## OPTIMIZATION STUDIES OF VARIOUS COAL CONVERSION SYSTEMS

WEST VIRGINIA UNIVERSITY  
DOE - \$208,173; WVU - \$13,878  
3/76 - 2/79  
Principal Investigator - C.Y. Wen

**OBJECTIVES** – A generalized standard method of comparison will be established employing mathematical optimization techniques which can be utilized in evaluating each unit operation found in various coal conversion processes. The work will provide a critical assessment of coal conversion processes and produce system models in identifying those processes or process steps that are major technical bottlenecks. This is done by considering, within existing physical, technological, and environmental constraints, various coal conversion alternatives and by employing cost minimization criteria as the basis for the selection of the most commercially attractive processes.

**RECENT WORK AND ACCOMPLISHMENTS** – A correlation for the formation of asphaltene through a consecutive first-order reaction mechanism has been developed. The concentration of asphaltene is related to the reactor residence time, coal dissolution rate, and the ratio of volumetric concentrations of asphaltene and moisture-ash-free coal in the feed slurry. Using the correlation developed for asphaltene formation and the experimental asphaltene-viscosity relationship in Synthoil liquid product, the variation of viscosity pattern in the reactor exit can be predicted with fairly good agreement. It is postulated that asphaltene content would reach a point beyond which successful reactor operation would be hindered. A computer model was established to simulate up-flow and down-flow in entrained bed gasifiers. Computation results are in agreement with pilot plant tests of the Texaco Synthesis Gas Generator. The Texaco process utilizes high ash H-coal residues from liquefaction process which are gasified with steam and oxygen to produce hydrogen and carbon monoxide. This computer model employs numerical techniques to solve the

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simultaneous heat and material balance equations for the complex reaction kinetics of pyrolysis, char-combustion, char-steam reaction, char-CO<sub>2</sub> reaction, char-hydrogen reaction, and water-gas-shift reaction. This model provides both temperature and concentration profiles for solid and gas phases.

**PLANS FOR THE COMING YEAR** — The current investigation on coal liquefaction study will be extended to include the effect of operating parameters on process thermal efficiency, feasibility, and economic evaluations for alternate conversion technologies. Work on entrained bed gasifier modeling will include investigations of more accurate reaction rate expressions, studies of hydrodynamics and mixing effects, and testing the limitations of the model. Efforts will be aimed at the development of a sophisticated model that is more compatible with existing pilot plant results.

## METAL CATALYZED REACTIONS OF POLYAROMATIC COMPOUNDS

UNIVERSITY OF WISCONSIN, MADISON

DOE - \$40,000; UW - \$3927

8/29/77 - 8/28/79

Principal Investigator - P.M. Treichel

**OBJECTIVES** — This project will study routes of conversion of polyaromatic substances and, ultimately, the conversion of coal itself, to simpler useful molecular products. The thesis for this work is that by coordinating a transition metal to an arene (here, as part of a polyaromatic compound), the arene reactivity can be specifically altered to give desired reaction sequences and useful products. The results of this preliminary study can be applied to the more practical goals of this project, the conversion of coal to important industrial chemicals.

**RECENT WORK AND ACCOMPLISHMENTS** — Assembly of equipment and supplies and a preliminary study on the syntheses of several complexes of polyaromatic complexes have begun. Some success has been achieved in this process, with a series of compounds being prepared.

**PLANS FOR THE COMING YEAR** — Initial work in this project will necessarily be concerned with the synthesis of metal complexes of polycyclic aromatic hydrocarbons, ML<sub>n</sub>(aromatic). Specific interest will be on varying metal and auxiliary ligands, the ML<sub>n</sub> group, since the reactivity of these complexes should be dependent on these parameters. Examples to be investigated include Cr(CO)<sub>3</sub>(aromatic), [Mn(CO)<sub>3</sub>(aromatic)]<sup>+</sup>, [Fe(C<sub>5</sub>H<sub>5</sub>)(aromatic)]<sup>+</sup>, and [Ru(C<sub>5</sub>H<sub>5</sub>)(aromatic)]<sup>+</sup>. Following the synthesis of these complexes, there will be a study of their reactivities. A wide search will be conducted for types of reactions, assessing the influence of the metal substituent in these chemical processes. Particular interest will be directed to oxidation and reduction reactions in these systems, substitution reactions, and reactions involving skeletal rearrangements or fragmentations. Some of these processes may prove to be a direct consequence of the metal substituent groups. A survey of possible reactions is the first step in identification of this phenomenon.



## PARTICULATE REMOVAL BY SELF-AGGLOMERATION IN A CYCLONE

UNIVERSITY OF WISCONSIN, MILWAUKEE

DOE - \$40,000

9/1/77 - 8/31/79

Principal Investigator - K.C. Tsao

**OBJECTIVES** – This program studies high-temperature and/or high-pressure particulate removal through a multi-inlet, multi-pass cyclone with in situ combustion for potential application in a combined gas-steam turbine cycle operating jointly with a pressurized fluidized-bed power plant. Other objectives are to conduct an experimental test on the proposed new particulate removal mechanism, to emphasize the practicability of the hardware design, and to verify the technical and economical feasibility on future utilization.

**RECENT WORK AND ACCOMPLISHMENTS** – A preliminary mathematical model is analyzed and partly completed, describing the probability of particle collision and agglomeration phenomena by reheating the fluidized-bed combustion products near coal-ash fusion temperature. Prediction of the size of the entrapped solids after agglomeration and their number density can be estimated based on the size composition of incoming dust-laden gases and the jet velocity profile.

**PLANS FOR THE COMING YEAR** – Work will include an attempt to modify the present mathematical model to identify the engineering parameters which would accelerate the agglomeration process in a cyclone with in situ combustion. Hardware design of an experimental high-temperature cyclone bench set-up and testing will be the main goal of this research.

## SELF-STUDY UNITS ON ENERGY FOR HIGH SCHOOLS

UNIVERSITY OF WISCONSIN, WHITEWATER

DOE - \$10,831

1/1/77 - 12/31/77

Principal Investigator - C.W. Shinnars

**OBJECTIVES** – A 1-week workshop on Energy at the University of Wisconsin, Whitewater was conducted during the week of August 8 to 12, 1977. Each of the participants was required to create a self-study unit on an energy topic to be used by high school students. The ultimate objective of the program was to increase the energy education level of 25 high school teachers and to develop a collection of energy self-study units.

**RECENT WORK AND ACCOMPLISHMENTS** – The workshop was conducted as planned, and 26 self-study units were produced. The units were edited by the program directors and printed collectively with copies being redistributed to each of the participants.

**PLANS FOR THE COMING YEAR** – The first phase of the program has been completed.

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## REMOTE ELECTROMAGNETIC SENSING OF UNDERGROUND COAL BURNS

UNIVERSITY OF WYOMING  
DOE - \$147,413; UW - \$40,570  
6/1/76 - 5/31/78  
Principal Investigator - E.A. Quincy

**OBJECTIVES** — This project seeks to develop and field test an electromagnetic induction system and estimator algorithm for remote determination of the location, shape, and size of underground coal burn voids. The information gained from this research will determine how efficiently the underground coal seam has been utilized in the production of gas.

**RECENT WORK AND ACCOMPLISHMENTS** — Two different configurations of wide-bandwidth induction systems were field tested at the Hanna underground coal gasification site of the LERC. The first system employed pseudo-noise transmitter waveforms and a cross-correlation receiver operating over a 0 to 50 kHz bandwidth to obtain time domain signatures of the underground coal burn in the field. Horizontal 1-meter loops were employed with 9 transmitting turns and 48 receiving turns. The second system operated over a 0 to 10 kHz bandwidth with a 220 turn receiving loop to shift more power to low frequencies for better depth penetration. Time responses from the field data were processed on the Fourier analyzer system and three-dimensional response maps produced in time and frequency domains. Strong anomalies at 5 kHz were observed along traverses on both burns at the Hanna II site. These anomalies correlated well with the location of the burn region. Computer programs for generation of induction responses of buried conducting bodies have been written and tested. Models have included rectangular boxes and spheres. The effect of conducting wells added at each end of a buried box was insignificant. The effect of the air/earth interface at the loops was shown to be only of secondary importance. Bayes statistical estimator algorithms were developed in closed form to operate on noisy induction responses for one or two parameters of buried conducting model bodies. These minimum mean-square-error estimators were shown to be unbiased and closed form expressions were developed for the mean-square-error and variance of the estimators. The estimators were shown to work well on simulated noisy induction responses obtained from the above computer programs with random noise added.

**PLANS FOR THE COMING YEAR** — Core samples will be obtained from the LERC for one of the burns at Hanna II. The conductivity of these samples will be measured to determine the electrical properties of the site after burning. These measurements will be used to more accurately represent computerized induction models and to correlate with field measurement induction anomalies. Estimator algorithms will be applied to the induction field data to determine the size and shape of the coal burn regions.

## EFFECTS OF SOLVENT CHARACTERISTICS ON WYODAK COAL LIQUEFACTION

UNIVERSITY OF WYOMING  
DOE - \$234,900; UW - \$32,214  
5/1/76 - 7/31/78  
Principal Investigators - H.F. Silver, R.J. Hurtubise

**OBJECTIVES** — This research investigates the effects of both gross and specific solvent characteristics on the extent of Wyodak coal liquefaction, asphaltene formation, and nitrogen removal during the noncatalytic hydrogenation of Wyodak coal. This work could be of potential interest in donor solvent and SRC II type processes. Successful completion of this objective would

provide a basis for improving coal liquefaction processes by indicating what process modifications, if any, could be made on coal-derived recycle oils used to slurry reactor feed coals.

**RECENT WORK AND ACCOMPLISHMENTS** – In the initial phase of the project, a Wyodak coal-derived recycle solvent from Wilsonville, Alabama was used to dissolve Wyodak coal in a magnedrive autoclave reactor. Results suggest that more attention could profitably be directed toward mass transfer considerations in the design of full-scale, flow system coal liquefaction reactors. In the current phase of the program, duplicate coal liquefaction runs have been made using 13 different coal-derived liquid solvents. All solvents boiled below 1000°F. Preliminary attempts to correlate the extent of liquefaction data using gross characteristics of the solvents suggested that two parameters may be required to define the solvent: a physical parameter and a chemical parameter. The analytical technique involved open column chromatograph with  $\text{Al}_2\text{O}_3$  followed by high-performance liquid chromatography to separate compounds. Individual compounds were then identified using fluorescence spectroscopy. No correlation was found between the extent of Wyodak coal liquefaction and solvent tetralin content. Total nitrogen removal from the Wyodak coal and the solvent used appears to decrease slightly with increased solvent boiling range. However, preliminary results suggest that for a solvent with a fixed nonpolar solubility parameter, the extent of coal liquefaction increases significantly with the solvent boiling range. In additional studies directed toward the effect of solvent on nitrogen removal, nitrogen classes were identified in three different solvents and in nine different solvent refined coal (SRC) products using nonaqueous potentiometric titration and infrared analysis.

**PLANS FOR THE COMING YEAR** – Coal tar solvents from U.S. Steel, from the in situ coal gasification process in Hanna, Wyoming, operated by the Laramie Energy Research Center, and the Kentucky coal-derived SRC I solvent from Tacoma, Washington, will be hydrogenated to varying degrees. These modified solvents will then be used to liquefy Wyodak coal. This could provide a wider range of solvent characteristics for correlation purposes. Attempts will be made to extend analytical techniques to quantitatively identify tetralin in coal-derived solvents to higher boiling hydrogen donor compounds. Because phenols could also have a catalytic effect on coal liquefaction, attempts will also be made to separate and quantitatively identify monophenols in the coal-derived solvents.

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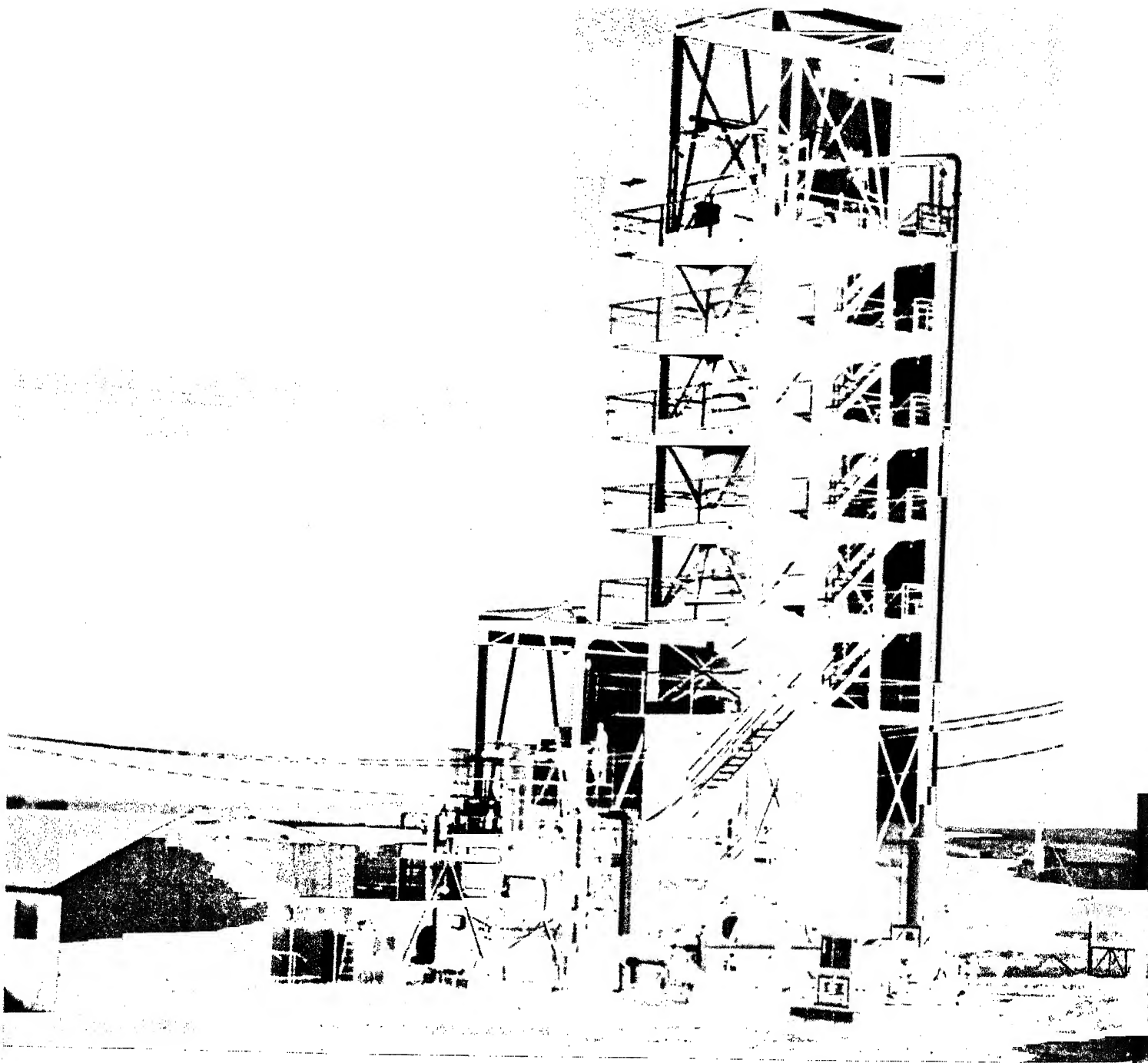
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*Laramie Energy Research Center 150-Ton Oil Shale Retort*

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## *ENHANCED OIL RECOVERY*

This program of research, development, and demonstration (RD&D) for enhanced oil recovery includes drilling and offshore technology and oil processing and utilization. The program involves cost-shared contracts with industry, in-house research at DOE Energy Research Centers at Bartlesville, Oklahoma (BERC); Morgantown, West Virginia (MERC); and Laramie, Wyoming (LERC). Projects also are conducted at Oak Ridge National Laboratory (ORNL), Los Alamos Scientific Laboratory (LASL), Sandia Laboratories (SL), Lawrence Livermore Laboratory (LLL), and Lawrence Berkeley Laboratory (LBL). Supporting research is performed at numerous universities. Rapid technology transfer is emphasized and is effected through periodic symposia, quarterly contract reports, in-house quarterly research reports, and technical presentations and publications by both DOE and contractor personnel.

DOE now has 22 cost-shared contracts with industry for demonstration, on a significant scale, of enhanced oil recovery (EOR) technology. The total cost of the multiyear contracts is \$156.6 million, of which industry is funding 64 percent. The program also includes field tests and supporting research at the institutions listed above, and at numerous universities and state agencies. EOR technology includes chemical flooding (micellar-polymer), thermal methods (in situ combustion and steam drive), carbon dioxide flooding, and improved waterflooding (polymer and caustic solutions). Goals for 1985 are the addition of 3 billion barrels of oil to proved reserves and an incremental daily production increase of 800,000 barrels.

The drilling and offshore technology program encompasses the following contracts: design and development of high-performance drill bits utilizing the Stratapax cutting element; use of mud-pulse telemetry in drilling; field test demonstration of Electrodril downhole drilling system; effect of offset angle in cutting, using a single roller cone; design of an improved pressure coring system with reduced core flushing; deep drilling simulation; downhole drilling motors study; sea floor instrumentation; offshore data acquisition and dissemination; and OCS analytical model. Major contracts are cost-shared with industry. A "blueprint" has been developed for a comprehensive Federal drilling technology-development program.

The oil processing and utilization program includes in-house research projects at Bartlesville and Laramie Energy Research Centers on oil-spill identification, characterization of heavy ends of petroleum, quality of crude oils and products, refining-process technology, and stability of hydrocarbon fuels. A contract also is included on refining characteristics of shale oil.

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## IMPROVED OIL RECOVERY BY SECONDARY AND TERTIARY METHODS

BARTLESVILLE ENERGY RESEARCH CENTER  
DOE - \$750,000  
1972 - Continuing

**OBJECTIVES** – This project is being conducted to improve waterflood technology by evaluating improvement in oil production of reservoirs in the midcontinent area from application of micellar-polymer techniques to successful waterflood projects. New technology that uses chemicals miscible with reservoir fluids and mobility-control agents for improving displacement efficiency can improve recovery from previously waterflooded reservoirs. Laboratory studies are necessary to determine rock, fluid, and chemical properties and their relationship to micellar slug size, composition, stability, and displacement efficiency. Field demonstrations are required to evaluate adsorption of the chemicals on reservoir rocks and degradation of the polymer as it is displaced through porous and permeable formations. The economics of this method also must be evaluated.

**RECENT WORK AND ACCOMPLISHMENTS** – Polymer injection was initiated in February 1977 at a rate of 80 bbl/d and an initial concentration of 1350 ppm. Designed reductions in viscosity after each 0.05 PV of polymer injected have been maintained, with current injection fluid concentration now measuring 620 ppm. Pressure surveys were made in all site wells to determine fluid balance within the pattern. An additional observation well to monitor flood progress has been drilled, completed, and is being sampled routinely. No increase in oil production has occurred to date. A project status report was presented at the ERDA Symposium, August 30-31. A presentation describing the automatic blending and injection system was given at the ASME Energy Technology Conference in Houston, Texas, September 18-22, 1977. Laboratory studies of the effects of mixing order in micellar formulations on viscosity, stability, and recovery efficiency have been conducted. Techniques for analyzing sulfonates in produced fluids have been developed and are used routinely in field analyses.

**PLANS FOR THE COMING YEAR** – Polymer injection will continue with a scheduled decreasing viscosity. Analyses of produced fluids for chemical breakthrough will be performed. History matching of the reservoir performance will be accomplished, with analyses studied to closely track recovery efficiencies.

## EL DORADO MICELLAR-POLYMER FLOOD

CITIES SERVICE OIL COMPANY  
DOE - \$5,392,490; Cities Service Oil - \$7,670,077  
6/17/74 - 11/82

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**RECENT WORK AND ACCOMPLISHMENTS** – Injection of micellar fluid formulated by Union Oil Co. commenced in the south Hegberg pattern of the El Dorado Field, Kansas, in October 1976, with reduction in injectivity occurring shortly thereafter. Attempts to remedy the situation involved stimulation with acid treatments, improved water quality, preheating the injection fluid, and filtration using DE filters. A pronounced improvement was achieved with the addition of a hydrocarbon solvent to the base crude used in the formulation. Tests indicate that a wax was being precipitated on the sand face as a result of a low-temperature zone downhole through which the fluids passed.

Micellar injection into the north Chesney pattern commenced on November 11, 1977. This fluid designed by Shell Oil Co. incorporates a biopolymer as part of the slug formulation for mobility control within the slug. Injectivity is being maintained at about predicted rates. The flood progress is being monitored with fluid sampling and logging at observation wells in each pattern.

**PLANS FOR THE COMING YEAR** – Injection will continue in both patterns. At present injection rates, the Union slug should be completed in November 1978, with the Shell slug injected by July 1978. A polyacrylamide mobility control buffer will immediately follow the Union slug. A biopolymer–polysaccharide–will be utilized behind the Shell slug. Process monitoring will continue to analyze test performance.

#### **BELL CREEK MICELLAR-POLYMER PROJECT**

GARY OPERATING COMPANY  
DOE - \$2,519,500; Gary Operating - \$2,519,500  
7/1/76 - 6/30/81

**OBJECTIVES** – This field pilot project is designed to demonstrate the feasibility of recovering tertiary oil from a successfully depleted waterflood by using a micellar-polymer flooding process and to extend the test results to the entire field. In addition, it might indicate what maximum recovery efficiency can be expected in field use of micellar-polymer floods. The pilot is a contained 40-acre 5-spot in a representative watered-out part of the Unit "A" reservoir of the Bell Creek Field, Powder River and Carter Counties, Montana. The total recovery from the pilot area is estimated to be 91,000 bbl. Expansion of the process to the full Bell Creek field could return an additional 54 million bbl after waterflood and will have application to similar Montana-Wyoming sandstone reservoirs.

**RECENT WORK AND ACCOMPLISHMENTS** – Extensive reservoir studies, site and pattern selection, design and selection of an optimal micellar-polymer system, history matching and preliminary process simulations by numerical models, and development of pilot injection and production wells were completed.

The major effort during this time was the design for the Bell Creek pilot of two optimal micellar-polymer processes, one oil-external and one water-external; and the concomitant development of a methodology for selecting the more suitable process by means of a standard set of laboratory experiments and numerical simulations. This effort was completed with the selection of the oil-external process. The results of the standard laboratory test series and the numerical simulation work appear to indicate that the superiority of the oil-external process results from its more effective displacement efficiency, mobility control, and protection against divalent ions.

Special note should also be taken of the effectiveness of the high pH silicate preflush in reducing adsorption and removing harmful divalent ions.

**PLANS FOR THE COMING YEAR** — Reservoir definition work will continue with tracer, mini-injectivity, and pressure pulse diagnostics, in addition to and iterating with ongoing numerical simulation work. The purpose of this work is to indicate that the reservoir site description is sufficiently accurate to lend confidence in the design and interpretation of the pilot performance.

Finalizing of design and construction of the injection plant and fluid supply logistics depended upon the details of the micellar-polymer process that was selected in August 1977. Plant design and construction are now being carried forward so that the mini-injection test can be made in the first quarter of 1978. The preflush, the soluble oil slug, and the polymer mobility control buffer will be injected during the first half of 1978. Injection of the tapered polymer slug continues for 18 months, until November 1979. Tertiary pilot peak-oil production should occur in the first half of 1979.

A field laboratory will be established as part of the injection plant, and there will be an ongoing monitoring program of produced fluid composition and properties, and of quality control for supplied chemicals.

### **WILMINGTON MICELLAR-POLYMER PROJECT**

CITY OF LONG BEACH, CALIFORNIA  
DOE - \$3,500,000; City of Long Beach - \$3,500,000  
7/6/76 - 12/31/80

**OBJECTIVES** — The objectives of this field pilot test are to demonstrate the technical and economic feasibility of recovering additional viscous crude oil from a watered-out, highly porous, unconsolidated sandstone reservoir by using a micellar-polymer process, and to extend the test results to full-field development. The pilot area in the Wilmington Field encompasses about 12 acres, 56 feet of sand pay, and a nominal 10-acre staggered line drive pattern of four injectors backed up against the Pier A fault. The oil gravity is 18 degrees API, and the viscosity 28 cp at reservoir conditions. It is estimated that a minimum of about 600 million bbl of oil can be recovered by micellar-polymer flooding from the approximately 1.3 billion bbl of oil left in the upper four zones of the Wilmington Unit. A similar amount, 600 million bbl of oil, is potentially available from the Long Beach Unit.

**RECENT WORK AND ACCOMPLISHMENTS** — Phase A, Preliminary Systems Design and Pattern Optimization, has been completed; Phase B, Advanced Systems Design, Site Preparation, Mini-Injectivity Test, and Engineering and Analysis, is nearing completion; Phase C, Systems Design Optimization, Well Preparation, and Plant Construction, is well underway; and Phase D, Preflush, Micellar-Polymer Process, Analysis, and Documentation, is pending completion of Phases B and C. Under Phase A, the laboratory testing for comparative analysis of the effectiveness of various sulfonates and polymers was initiated and actual reservoir data were used for pattern optimization via model simulation. Phase B has determined the composition of the micellar/polymer systems to be used, and contracts have been let for chemical procurement. A sulfonate suitable for fieldwide application has been developed from Wilmington crude oil.

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**PLANS FOR THE COMING YEAR** — Phases B and C should be accomplished by the end of the fiscal year and the preflush and micellar slug injection completed. A single well tracer test for determining residual oil is planned for late FY 1978.

### **PENN GRADE MICELLAR DISPLACEMENT PROJECT**

**PENNSYLVANIA GRADE CRUDE OIL ASSOCIATION**  
DOE - \$2,220,000; Penn Grade Participants - \$2,220,000  
6/30/75 - 6/30/80

**OBJECTIVES** — The Penn Grade Micellar Displacement Project is designed to demonstrate the efficiency and economics of recovering tertiary oil. The project is a 24-acre field demonstration test that contains nine 5-spot patterns. The oil recovery technique is a micellar-polymer displacement process for recovering residual oil from depleted waterfloods. The Bradford Oil Pool in New York and Pennsylvania contains over 80,000 acres under waterflood, most of which is nearing depletion in the low-permeability Bradford Third Sand Reservoir, which produces crude oil used in the manufacture of high-quality automotive and industrial lubricants.

**RECENT WORK AND ACCOMPLISHMENTS** — Forty-one wells were drilled and completed for the test pattern, and surface facilities were constructed. Core and log data obtained during the development were used in the laboratory to plan and design the oil recovery process. This project will involve the injection of a brine preflush (10 percent of a pore volume) to condition the reservoir after waterflood, a micellar solution (7 percent of a pore volume) designed to optimize the displacement of residual oil from the reservoir, and a polymer solution (50 to 100 percent of a pore volume) to protect the recovery efficiency of the micellar solution. After polymer injection, water will be injected to complete the process. The brine preflush has been completed, and the micellar solution is currently being injected into the reservoir. A program to monitor the injectivity and productivity histories is being conducted for use in the evaluation of this oil recovery project.

**PLANS FOR THE COMING YEAR** — It is anticipated that the micellar injection will be completed in the first quarter of 1978 and that polymer injection will then begin.

### **TERTIARY RECOVERY OF OIL WITH SURFACTANT SOLUTIONS**

**THE PENNSYLVANIA STATE UNIVERSITY**  
DOE - \$1,000,000; Penn State University - \$50,000  
2/1/75 - 1/31/80

**OBJECTIVES** — Specific emphasis has been placed on the design and development of surfactant solutions and polymer buffers for use in low-permeability Pennsylvania oil sands. An improved understanding of the interaction between the chemical flood solutions and the system in which they must function is being developed by fundamental studies as well as long-core tests. Use and/or disposal of the produced water for EOR is also being evaluated.

**RECENT WORK AND ACCOMPLISHMENTS** — A surfactant synthesis method has been developed by sequential chemical processes of vapor phase oxidation and sulfonation of paraffinic and naphthenic hydrocarbons. Three new surfactants have been prepared and characterized. In water solutions, they were found to have low interfacial tensions ( $\approx 10^{-3}$ ) with hydrocarbons over a wide range of n-paraffin chain lengths ( $C_6$  to  $C_{10}$ ), behavior that differs significantly from commercial

petroleum sulfonates. Several chemical flood systems have been designed and tested in long cores with these surfactants. High-concentration surfactant slugs have been prepared using Xanthan gum for the polymer buffer. A slug containing 0.5 percent surfactant for low-interfacial tension and 500-ppm Xanthan concentration for viscosity control has been core tested with excellent results for EOR. It produces equal or larger amounts of oil than does 4 to 10 times as much surfactant and polymer used in conventional high-concentration slugs.

Inaccessible pore volume effects in cores of reservoir rock have been elucidated by the measurement of polymer elution volume as a function of polymer weight and rock permeability. The data indicate that equilibrium distribution of polymer molecules is the controlling mechanism. For measuring the solution properties of Xanthan gum, a precision low-shear capillary viscometer has been designed and built to evaluate the molecular size (by intrinsic viscosity) and non-Newtonian flow at shear conditions in the range of  $<10$  to  $500 \text{ sec}^{-1}$  shear rate. The effects of orifice and propeller blending of Kelzan on molecular dispersion, filterability, mechanical degradation, and viscosity level have been determined, and water-polymer blends show the same trends that are well established for polymers used in organic solvents typical of automotive lubricants. These studies include polyacrylamide, colloid (polysaccharide) and Natrosol blends in water, and show good correlation with molecular size as measured by intrinsic viscosity.

Injectivity of Xanthan gum solutions has been evaluated as a function of polymer solution preparation and processing. Diatomaceous earth filtration preliminary to injection appears to be a necessary step to remove particles down to submicron size for injection into low-permeability (Pennsylvania) oil sands. Porous media viscometry techniques are being used to measure flow of both polymer and micellar solutions (slug). Behavior in the oil sand parallels that in the filter elements studied in the porous media viscometer. The mathematical model of three-component, two-phase, one-dimensional flow has been completed, the first computer code has been written, and is being tested. Low-flow-rate runs have been accomplished in 2- and 4-ft Berea cores that show an increase in tertiary oil recovery at very-low-flow rates caused by the greater mobility control of the buffer. The sweep efficiency studies cover buffers of varying viscosities. Displacement in both linear and areal models showed that the sweep efficiency depends strongly on buffer viscosity.

The investigation on the adsorption of Xanthan gum shows that the adsorption is particularly strong when the surfaces are positively charged. Addition of a salt tends to increase adsorption, whereas multivalent anions reduce it. Produced water from tertiary oil recovery in Bradford No. 3 oil sands may contain about 5000-ppm total salts, of which perhaps 30 percent are bivalent metal chlorides.

**PLANS FOR THE COMING YEAR** – Surfactant synthesis work will optimize the yield of monosulfonate of the equivalent weight predicted from the petroleum fraction feed to the vapor phase oxidation. Physical properties of these surfactants will be studied and the results used to design conventional and low-concentration surfactant floods in long-core tests. To tabulate fluid properties for application in EOR over a wide range in permeability, polymers used in buffer solutions will be studied. Flow studies will focus on developing a simple, precise, and reliable porous media flow test based on throwaway porous media. Mathematical modeling of the micellar flooding of the oil reservoir will be continued. Core testing will be extended to include the use of Pennsylvania oil sand. Low-flow-rate core tests will be continued using chemical flooding systems designed around surfactants.

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## NORTH BURBANK UNIT MICELLAR-POLYMER FLOOD

### PHILLIPS PETROLEUM COMPANY

DOE - \$3,402,042; Phillips Petroleum - \$6,352,472  
5/12/75 - 10/1/79

**OBJECTIVES** – This project is attempting to demonstrate that petroleum recovery can be increased by employing a well-designed tertiary recovery method to a currently producing reservoir that is approaching the economic limit of waterflood recovery. The Burbank reservoir in Osage County, Oklahoma is the field pilot used here. This reservoir is oil wet, so that a new technology had to be developed that should be applicable to a significant number of other fields.

**RECENT WORK AND ACCOMPLISHMENTS** – Polymer has been injected into Tract 97 in six concentration steps from 2000 ppm to the current 250 ppm. Laboratory analyses have provided constant monitoring of the polymer concentration and viscosity and the production of sodium, calcium, chloride, sulfonate, alcohol, and polymer. Wellhead pressures were followed to evaluate the mobilities of the chemical slugs, detect fractures, and correct fluid drift across the tract. Remedial work was performed on wells as needed: acidizing, frac treatment, and gelled polymer treatment. Tertiary oil is now being produced at a rate of 193 bbl/d and a WOR of 55, compared with an initial WOR of 200. Response was first noticed in the central 5-spot, and has climbed steadily as the outer wells began to respond.

**PLANS FOR THE COMING YEAR** – Polymer injection will be completed in January. A fresh water buffer will be injected for 2 months, followed by a produced-water drive. Reservoir engineering studies will be continued to indicate the need for flow-rate adjustments and well treatment, and to get an evaluation of project performance. Although the contract terminates in October 1979, reporting will continue for 1 additional year.

## COMMERCIAL-SCALE DEMONSTRATION MICELLAR-POLYMER FLOOD

### MARATHON OIL COMPANY

DOE - \$14,000,000; Marathon Oil - \$29,560,000  
9/30/76 - 12/31/86

**OBJECTIVES** – The objective of this project is to evaluate the technical and economic viability of the micellar-polymer flooding process for improved oil recovery. Because the 407-acre demonstration comprises both 2.5-acre and 5.0-acre well spacing, the effect of pattern size on economic recovery will be evaluated.

**RECENT WORK AND ACCOMPLISHMENTS** – Injection of the crude oil sulfonate slug commenced in February 1977 and has remained essentially stabilized at about 3200 bbl/d. All producing and injection wells taking less than desired rates have been successfully stimulated with designed hydraulic fracture treatments. Pressure transient tests on selected injectors and producers have been made quarterly to assess the change in flow parameters caused by the slug injection. Two observation wells have been completed for fluid sampling and logging to monitor flood front advance. Two reports were drafted describing the process design, engineering, and facilities used in this test.

**PLANS FOR THE COMING YEAR** – Micellar fluid will be injected into both 2.5-acre and 5.0-acre patterns until about April 1978, at which time the required volume will have been injected into the former pattern. A mobility buffer spike will be initiated and continued during the remainder of the year. Micellar injection into the 5.0-acre pattern will continue until mid-1979.

### LARGE-SCALE SULFONATE EOR STUDIES

SUNTECH, INC.  
DOE - \$390,208  
4/26/77 - 6/25/78

**OBJECTIVES** – This project is designed to provide large-scale, readily available samples and information that will help resolve questions/problems on the nature and supply of oil recovery sulfonates via the following tasks: review current refinery and petrochemical operations to select candidate feed stocks for the preparation of synthetic and petroleum sulfonates; study alkylation and sulfonation reactions and their products on a laboratory scale and survey the properties of these sulfonates in oil recovery systems; select a suite of samples from the candidates and prepare 50-lb lots of each (this quantity will be distributed in laboratory-sized samples to interested organizations); and provide refinery characterization data on all feedstocks used in the preparations; carry out a preliminary evaluation of these samples with respect to salinity, temperature, and oil recovery; extend analytical methods by which the range and average value of equivalent weight of synthetic sulfonates can be accurately measured; and have all matters (such as sample distribution, information input, and distribution) handled by an independent organization—the New Mexico Petroleum Recovery Research Center (Dr. J.J. Taber).

To realize the maximum benefit from this program, it is hoped that the sharing of non-proprietary information as expeditiously as possible will facilitate research on enhanced oil recovery.

**RECENT WORK AND ACCOMPLISHMENTS** – Preparation and planning for the production of the large-scale samples in the pilot plant at the Marcus Hook refinery is in progress. This effort has been directed toward the synthetic sulfonates. The aromatic component will be derived from the common BTX (benzene-toluene-xylene) operations. For the olefins in the first group, the refinery/petrochemical streams chosen are the propylene tetramer and pentamer. The options for the alkylation reaction include the use of  $\text{AlCl}_3$  as catalyst in plain or modified form. As more varied structures were examined in this study, the reaction parameters were increasingly well defined in problem areas such as side-chain degradation.

A program was pursued for upgrading analytical methods, especially characterization of polysulfonation by thin-layer chromatography or high-pressure liquid chromatography. ASTM methods for sulfonate determination are not satisfactory on the high-molecular-weight substances of interest. Characterization of the sulfonates was made by phase studies and recovery tests. Results with the middle phase-optimal salinity approach gave the following indications: some samples, especially where interfacial tensions are very low, take a very long time to reach equilibrium—changes are still occurring after 3 months; effective recovery systems were found in which oil solubilization is very low, even though a middle phase is present; some cases were observed in which the sulfonate was at an appreciable concentration in two phases rather than just one; examples were observed in which alcohol distribution was over two or three phases rather than just one.

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A questionnaire was sent to interested parties in September, with the expectation of finalizing the list of petroleum sulfonates to be made available for distribution by October 1977.

**PLANS FOR THE COMING YEAR** – Production of the large-scale sulfonate samples and their distribution to interested parties should be completed as should the evaluation of the properties of these samples and the improvement of analytical techniques. Requests for special small-scale samples will be filled, and the major portion of the information/data processing should be accomplished.

### HUNTINGTON BEACH ALKALINE FLOOD

AMINOIL USA  
DOE - \$499,400; Aminoil USA - \$1,475,000  
7/1/77 - 7/1/79

**OBJECTIVES** – This project seeks to determine the current oil saturation at the pilot site, to design a field-specific alkaline flooding process, and to test the process in a pilot flood. Many California reservoirs are good candidates for alkaline flooding. The crude oils involved are acid and are also somewhat viscous for good waterflood recovery, although the reservoirs are generally waterflooded. This project can provide further information on alkaline flooding mechanisms for the national EOR program and develop enhanced oil recovery in the Huntington Beach field.

**RECENT WORK AND ACCOMPLISHMENTS** – Design work on the alkaline flooding process is well underway. Laboratory work has been directed at optimizing the size and salinity of the soft water preflush; the size, salinity, and alkaline concentration of the chemical slug; and the size and salinity of the soft water after flush. Plans are to use 40 percent of a pore volume (PV) softwater preflush; 40 percent PV of 0.15 weight percent alkaline slug, and 40 percent PV softwater after flush. Total injection period will be about 3.5 years at rates of 10,000 to 18,000 bbl/d. The alkaline chemical will be composed of three quarters sodium hydroxide and one quarter sodium silicate. The soft water used will be a combination of produced and brackish water, softened to remove calcium and magnesium, with a salt content of 7500 ppm. This water will also be used to make the alkaline slug.

The central injection well for the pilot test has been redrilled, logged, and cored. Sections of the core will be used for further laboratory tests on process design. The coring operation was done alternately with plastic sleeve or pressure core barrels. Pressure core recovery was reasonably good from the unconsolidated sands and is expected to provide actual values of in-situ porosity and saturations.

**PLANS FOR THE COMING YEAR** – Injection of the softwater preflush should begin early in 1978. After preflush injection a tracer test program will be run to determine pattern sweep efficiency. Alkaline slug injection will begin shortly after the tracer test is completed.



## IMPROVED OIL RECOVERY BY CONTROLLED WATERFLOOD, ALKALINE FLOOD

CITY OF LONG BEACH, CALIFORNIA  
DOE - \$4,633,829; City of Long Beach - \$6,950,743  
10/1976 - 9/30/81

**OBJECTIVES** – This project is a demonstration of caustic waterflooding in a typical well flood pattern of the Ranger Zone of the Long Beach Unit portion of the Wilmington Field, California. Two alkaline flood processes are to be used in this demonstration; entrapment process, in which caustic in softened fresh water is to be injected to improve sweep efficiency, followed by the entrainment process, in which caustic and salt are injected to improve flood displacement efficiency. The demonstration involves the input of 30,000-bbl/d water in eight injection wells that surround 11 active producers in an area of 95 acres. If successful, improved flood efficiency would prevent early abandonment of most wells because of high water-oil ratios and would increase oil recovery.

**RECENT WORK AND ACCOMPLISHMENTS** – The major emphasis during this period was on the well repair and redrilling necessary to conduct a satisfactory pilot test. The injection wells were found to be in poorer condition than anticipated, requiring redrilling in five cases and repair on two producers and one injector. Two wells were thermally stimulated over a portion of the Ranger interval. As planned, three injectors had dual injection tubing strings installed to permit better profile control of the injected water.

To evaluate the reservoir rock properties in the pilot area more accurately and provide new, representative core material for laboratory flood tests, two of the redrilled wells were cored. Special electric log suites were run in these two cored wells and one other redrilled well. The log suites were analyzed quantitatively to give another independent source of data on in-situ formation properties. Laboratory core flood tests were started to evaluate the caustic process. Running such tests with a high viscosity oil under overburden pressures involved a number of new procedures such as the collection of representative data on core resaturation techniques and flood rates.

Laboratory tests were made to establish the best method of removing caustic water from Ranger crude. This work indicated that dehydration would not present unusual or costly operating problems. A small-scale pilot test was started to evaluate methods for softening the field's saline produced water for use in future caustic injection projects.

Design of the additional facilities needed for the caustic injection were essentially completed. A short-term mini-injection test of a pilot area injector was conducted. A total of 236,748 bbl of softened fresh water with 0.1 percent by weight NaOH was injected at a 4500 to 5000 bbl/d rate. Samples of the well bore fluid and reservoir fluids backflowed at the end of the test were analyzed. They indicated that while some reduction in injectivity occurred, it did not continue to increase with time and was not of a magnitude that would prevent reaching planned injection targets.

**PLANS FOR THE COMING YEAR** – Cleanout and stimulation of one injector and installation of flow control equipment in another injector are anticipated. No further redrills are expected unless well conditions deteriorate unexpectedly.

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Preliminary core flood testing should be completed early in the period. Additional second- and third-phase flood tests also should be finished as should the produced water softening pilot test. Final design, procurement, and installation of additional caustic injection facilities will then proceed.

The matching of past waterflood history of the pilot area with the reservoir simulator and a projection of the area's behavior under a continuation of plain waterflooding will be made. Program modification to model the caustic process will be completed and expected caustic waterflood behavior will be projected. Initial preflush injection in the eight pilot area wells should start before the end of FY 1978, followed by a number of planned special preflood well tests and surveys. Other producing operations are expected to continue in a routine fashion during this period.

### **ATTIC AIR INJECTION PROJECT**

**OIL DEVELOPMENT COMPANY OF TEXAS**  
DOE - \$496,932; Oil Development - \$49,147  
4/1/76 - 2/31/78

**OBJECTIVES** – This project will demonstrate the technical feasibility and economics of oil recovery from a low productive reservoir, the Willow Draw Field in Park County, Wyoming, using the attic air injection process. This field originally contained 177 million barrels of 13 to 17 degree API oil. The attic air injection project is expected to increase ultimate recovery to 4 percent of the original oil-in-place from the estimated primary recovery of 1 percent.

**RECENT WORK AND ACCOMPLISHMENTS** – Mechanical difficulties with the compressors have been controlled by installation of a four-stage air compressor, and steady injection at 300 Mcf/d has been generally maintained. Air breakthrough problems at producing wells were controlled by automatic annulus backpressure relief valves set at 50 to 100 psig until September 1977. It was decided then that air control workovers were justified because field oil production decline had been reduced from 48 percent to 24 percent and because displacement of oil from the rock matrix was inferred from field performance. The net voidage since project start has increased 73,000 bbl, but field reserves have improved 160,000 bbl based on decline calculations. Currently, it is considered that the oil displacement process is working, but it is not determined if it will work in the long term at an economic rate.

**PLANS FOR THE COMING YEAR** – In the period September to December 1977, air injection will be suspended and ten structurally high wells will be reworked. In January 1978, the air injection rate will be increased to 455 Mcf/d and sustained at that level until air breakthrough is noted, then reduced to 300 Mcf/d. Subsequently, injection will be maintained at 300 Mcf/d until cumulative injected volume equals cumulative oil withdrawal. At that time, the air injection rate will be reduced to the equivalent of the stabilized oil withdrawal rate. Successive programs to lower the fieldwide perforation levels will be required in the post-contract period to determine if the oil displacement process will work over the long term at economic rates.

## NORTH STANLEY POLYMER PROJECT

KEWANEE OIL COMPANY

DOE - \$1,150,752; Kewanee Oil - \$2,734,148

7/1/75 - 5/31/79

**OBJECTIVES** – This polymer test at the North Stanley Stringer, an offshore sand bar, is designed to determine if oil can be economically recovered from a Bartlesville sand reservoir by injecting a polyacrylamide polymer during the mature stage of waterflooding, to gain a better understanding of the mechanics of polymer displacement, and to ascertain whether a polymer flood will enhance the probability of success of micellar flood by replacing the connate water with water containing lower concentrations of calcium and magnesium ions and by reducing the effect of high permeability variance. The project is considered to be a true field test as it encompasses 1010 productive acres and 72 million barrels of pore volume, utilizes the current producing and injection wells, and has a well-defined producing history that can be used as a baseline for judging project response. The North Stanley Stringer, located in Osage County near Shidler, Oklahoma, has a remaining reserve of 2 million bbl if the polymer injection program is modestly successful; in addition, this technique can be applied to similar reservoirs.

**RECENT WORK AND ACCOMPLISHMENTS** – The primary activity was the successful injection of the polymer slug on June 22, 1977, after injecting 1,194,770 pounds of Dow Pusher 700 and 11,962,918 barrels of water over a period of 372 days. Average polymer concentration during the period was 285 ppm. Mixing and injection equipment functioned extremely well and was operated by the lease pumper with a minimum of supervision. The primary producing problem experienced was a change in the producing well fluid levels resulting from the changes in injection distribution. Since most of the wells are produced through downhole centrifugal pumps that are designed for a specific fluid level, it was necessary to change out the pump or lower the present pump, which resulted in less efficiency. Although it is not possible to predict the magnitude and location of fluid level (i.e., reservoir pressure) changes in advance, anyone planning a polymer injection project should allow for corrections to be made in pumping equipment.

To accelerate the change in injection profiles and to decrease per well injection rates below 3000 bbl/d, nine of the injection wells were given Channelblock® treatments. One well, the closest to the injection plant did not respond to this treatment as the pressure was too high for the soft gel to hold. The rate was then restricted by use of a flow regulator coil. The polymer at 250 ppm concentration experienced a 12 percent shear degradation passing through the regulator coil. Movement of the slug through the reservoir was monitored by analysis of produced water samples for changes in salinity and the presence of polymer.

Oil production started responding to polymer injection in September 1976 by increasing from 15 to 581 bbl/d. The average oil production was 631 bbl/d for September 1977, which was 41 above the anticipated polymer response curve, and 146 above the estimated normal production decline curve. Tertiary oil production averaged 146 bbl/d for a monthly total of 4380 bbl. Cumulative tertiary oil production totaled 42,641 bbl. The average WOR has decreased from 76.5 in July 1976 to 52.0 in September 1977, indicating its response to the injected polymer. Although this response has exceeded predictions, production must continue to increase if the overall project goals are to be met.

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**PLANS FOR THE COMING YEAR** – A detailed study will be made to identify what changes have occurred in the fluid movement in the reservoir and how the movement will affect sweep efficiency and ultimate recovery. As a result of that study, a firm response projection will be made, and project economics will be calculated. In the immediate future, the feasibility of a micellar injection project will be determined. If the micellar project is not feasible, fresh water injection will be terminated and replaced with produced water injection.

## **COALINGA FIELD POLYMER FLOOD**

**SHELL OIL COMPANY**  
DOE - \$2,168,206; Shell Oil - \$5,206,666  
6/75 - 7/79

**OBJECTIVES** – This phase of the pilot demonstration project is designed to determine the waterflood base case for a multilayered reservoir prior to polymer injection. The reservoir contains a medium viscosity oil and has an unfavorable water displacement ratio. A successful project is expected to recover an additional 2.6 million barrels of oil from the pilot area, of which a polymer flood is expected to recover about 600,000 barrels. Expansion of the flood to other areas of the Coalinga Field is expected to recover 35 million barrels of oil, including 8 million barrels from polymer injection. The process would be applicable to other fields having reservoir characteristics similar to those of Coalinga Field.

**RECENT WORK AND ACCOMPLISHMENTS** – Fresh water, containing 300 ppm sodium chloride, has been continuously injected since mid-1976 at a rate of 1350 bbl/d into each of the four injection wells. Periodically, bottom hole pressures and the injection profiles have been obtained. A different tracer was injected into each of the injection wells, and the path of the tracers monitored. Oil well production, both within and outside the pattern area, has been closely monitored also. Stimulation treatments and remedial work have been performed to improve production characteristics. Water samples have been obtained and salinity data gathered. An extensive well abandonment program has been in progress to eliminate idle and nonpattern wells within the pilot area. Production from individual sands has been monitored in the production observation wells to interpret the flood performance. A dual induction log was run 42 times in the logging observation well and the changes in resistivity followed.

Additional laboratory tests and a series of polymer injection tests have been conducted in the area of the pilot project to evaluate the polymer slug design further. Modifications have been made to the polymer mixing and injection plant to improve plant efficiency. The petrophysical description of the reservoir has been updated from data obtained by additional core analyses and well logs. Production, injection, and petrophysical data are being incorporated into the computer program for matching and predicting the performance of the pilot area.

**PLANS FOR THE COMING YEAR** – The pilot area will continue to be waterflooded until a base case has been obtained. Data from the injection, production, and observation wells will continue to be gathered and analyzed. Well performance will be monitored, and programs to improve performance of wells will be initiated as required. Laboratory and polymer injection tests will continue to determine optimum polymer slug design. Petrophysical and production data will be used to further update the computer program for matching and predicting the performance of the pilot area. Polymer injection will be initiated upon obtaining a waterflood base case and the design of polymer slug design completed.

## CHARACTERIZATION OF RESERVOIRS FOR IMPROVED RECOVERY

BARTLESVILLE ENERGY RESEARCH CENTER  
DOE - \$152,000  
1976 - Continuing

**OBJECTIVES** – Knowledge of the characteristics of reservoirs is necessary to determine what type of enhanced recovery operation can be applied to them. Specifically, research will be performed to determine the characteristics of petroleum reservoirs that are the most susceptible to enhanced recovery operations; to determine what causes the salinity persistence effect in formation water and how to compensate for it; to determine the solubilities of compounds that form by the interaction of polyvalent ions in formation waters with chemicals used in enhanced recovery; and to develop standard methods for determining the quality and compatibility of waters injected into petroleum reservoirs and for determining environmental impacts.

**RECENT WORK AND ACCOMPLISHMENTS** – Oil and gas reserve data, reservoir depositional environment and lithology, type of trap, drive mechanism, porosity, permeability, reservoir temperature and pressure, salinity and related information were determined for the reservoirs in the Permian Basin. Data reduction, formatting, and keypunching continued in an effort to build an oilfield brine analysis data bank consisting of about 100,000 samples. Tolerances of commercial petroleum sulfonates at 3 percent concentrations to calcium in solutions at 30°C and ambient pressure were determined. A potentiometric method using a PVC membrane electrode to determine concentrations of sulfonates in aqueous solutions was developed. Standard methods to determine injection water qualities were published. An investigation of possible uses of oilfield waters in arid areas was completed. Locations of potential lithium reserves in oilfield brines were identified—lithium is needed for fusion energy.

**PLANS FOR THE COMING YEAR** – A report on characteristics of reservoirs in the Permian Basin for optimum application of enhanced recovery operations will be completed. The oilfield brine analysis data bank should be completed, edited, and put on line probably with the Petroleum Data System computer so that it can be used by the Government and private industry in studies related to production, enhanced recovery, exploration, and environment. Tolerances of divalent ions to commercial petroleum sulfonates at elevated temperatures and pressures will be determined. Research will be conducted to determine the causes and effects of the salinity persistence effect. Methods to determine injection water quality, compatibility, and sources will be developed. Characterization of another basin for EOR (probably the Gulf Coast Salt Basin) will be started after contract negotiations.

## MICELLAR PROPERTIES OF OIL AND ADDITIVES FOR INCREASED RECOVERY

BARTLESVILLE ENERGY RESEARCH CENTER  
DOE - \$90,000  
1975 - Continuing

increase crude oil production can easily produce harmful side process, knowledge of what materials in complex mixtures change he like is needed. This study seeks to determine how colloidal

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properties such as micellar size and shape are influenced by additives and by physical changes made to increase production, and how properties of such colloidal solutions as detergents and polymers are changed by contact with crude oil and by changes in their chemical composition and physical treatment.

**RECENT WORK AND ACCOMPLISHMENTS** – Several hundred small-angle X-ray scattering studies were made on whole and treated crude oils to determine the effects of addition of various solvents, such as hydrocarbons and alcohols, and to determine the effects of temperature variation. Three different types of colloids were found in crude oils (a few oils contain all three): the normal-sized asphaltic colloids, much larger colloids that seem to be associated with asphaltic materials in some manner, and waxy colloids of large size.

Accurate measurements of the densities of the detergent, sodium dodecyl sulfate (SDDS), in several background concentrations of NaCl (aq) and in water have been made. These data were used to determine the accurate partial specific volumes required for ultracentrifuge studies. An isopiestic distillation apparatus has been constructed and is being used to provide data for correction of the ultracentrifuge results. Ultracentrifuge experiments have been completed on solutions of SDDS in 0.1 m and 0.2 m NaCl (aq). Final interpretation of these experiments awaits the isopiestic distillation results.

**PLANS FOR THE COMING YEAR** – Ultracentrifuge and related studies of detergents in water and brine will be carried on. X-ray studies of the colloidal properties of oil will continue as well as some studies concerning polymers used in micellar flooding. A publication concerning the results of the X-ray studies of oils is likely.

## THERMODYNAMIC CHARACTERIZATION OF TERTIARY RECOVERY SYSTEMS

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$125,000

1/74 - Continuing

**OBJECTIVES** – This research program is investigating the phase and interfacial thermodynamic properties of surfactant systems as they relate to enhanced oil recovery processes. This work will provide practical yet fundamental knowledge about the thermodynamic behavior of surfactant systems under varying conditions of temperature, pressure, concentration, formation water, surface, and oil in place. The strength and type of interaction between injected fluids and reservoir materials are important parameters for successful enhanced oil recovery process design. Correlations of surfactant thermodynamic properties with micellar stability in oilfield reservoirs will be valuable for development of enhanced recovery techniques.

**RECENT WORK AND ACCOMPLISHMENTS** – The heat of micellization and dilution of sodium dodecyl sulfonate and sodium decyl sulfonate have been measured calorimetrically in a variety of salt and alcohol backgrounds. The enthalpy of micellization is extremely sensitive to temperature, salt background, and alcohol background. Large differences are observed between the results obtained for sodium dodecyl sulfonate and sodium decyl sulfonate. An adsorption calorimeter has been put into operation for measurements of heats of adsorption of surfactants on reservoir materials. Initial measurements have been made with sodium dodecyl benzene sulfonate onto silica

gel. Pure samples of sodium decyl benzene sulfonate and sodium dodecyl benzene sulfonate have been obtained.

**PLANS FOR THE COMING YEAR** – Enthalpy of micellization measurements will be made on sodium dodecyl benzene sulfonate and sodium decyl benzene sulfonate. High-temperature heat capacities will be measured at the University of Delaware under contract arrangement. A heat-capacity microcalorimeter for measurement of heat capacities near room temperature will be assembled. A densimeter for determination of partial molal volumes of surfactants will be operational, and measurements will be made on surfactant systems. Enthalpy measurements may be made on mixed micelle systems. All enthalpy results will be analyzed, and correlations with molecular structure will be attempted. A concentration detection system will be installed for use in adsorption studies. Adsorption measurements will be made with a high-purity surfactant on sand and clay surfaces. The effect of alcohols and salt on adsorption will be investigated.

## CHEMICALS FOR ENHANCED OIL RECOVERY

OAK RIDGE NATIONAL LABORATORY  
DOE - \$386,500  
4/23/76 - Continuing

**OBJECTIVES** – The objective is to find sources of raw materials in industrial-waste streams and in natural products for the production of chemicals for EOR. The emphasis is on lowering costs and on increasing availability by identifying materials that require relatively little adaptation for use and by finding alternatives to present procedures. Chemicals contribute a major part of the costs of EOR by micellar-polymer flooding; estimates range from \$4 to \$9 per bbl. In addition, large-scale chemical-production capacity is limited. Cheaper supplies and more assured availability would increase the attractiveness and decrease the risk of applying chemical procedures. It is projected that surfactant-based methods might contribute about half of the increased production from known U.S. fields by presently developed tertiary techniques, or perhaps 60 million bbl.

**RECENT WORK AND ACCOMPLISHMENTS** – Foam stabilization properties of a fraction obtained from weak black liquor of the kraft pulping process indicated the presence of a surface-active agent, but there is no indication that substances other than tall oils are responsible. Interfacial tensions (IFT) between hydrocarbons and aqueous solutions containing sodium oleate, as a model for tall oils, are under investigation for a range of surfactant concentrations. The effect of pH on IFT becomes smaller as the oleate concentration is increased. No conditions of salinity, pH, hydrocarbon, type of cosurfactant, etc., for which interfacial tensions are in the millidyne/cm range have been found.

Fermentation tests were made on samples of waste materials and inexpensive carbon sources such as starch, hydrolyzed sugar-beet pulp, hydrolyzed oat-straw, hydrolyzed corn cobs, and hydrolyzed white water fines (from wood pulping). Several small fermenters have been placed in operation for the production of 1-liter batches of single cultures. The 10-liter Chemapac fermenter is being used to produce polymers via continuous fermentation with *Sclerotium rolfsii*. Measurements have been started of the adsorption of glucan polymers from water on ground Berea sandstone. The adsorption appears somewhat higher than that of commercial xanthan gums. It also appears to have viscosity properties similar to commercial xanthan gum at comparable concentrations in terms of dependence on shear, temperature, pH, and salinity; it is, if anything, less

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sensitive to hardness. Since the fermentation proceeds under acid conditions, less difficulty with contaminating organisms might be expected. Although fermentation conditions were not optimized, encouraging yields of polymer were obtained in several instances. The highest polymer concentration reached was 18 gm/liter (over 30 percent conversion) with starch.

Evaluation of fix-film, upflow, anaerobic bioreactors for production of cosurfactants has begun. Substantial conversion of black strap molasses to butanol and other products by *clostridia* cultures has been effected. Attempts to adapt the organisms to produce the chemicals from wood molasses have not so far been successful.

**PLANS FOR THE COMING YEAR** — The present line of endeavor to produce EOR chemicals such as polymers, alcohols, and surfactants by chemical modification and/or biological action on waste materials will be continued. Emphasis will be placed on methods adaptable to scale up for carrying out microbiological production of polymers and other chemicals. Samples of any chemicals that appear of potential value in the preliminary screening will be submitted to outside laboratories for detailed evaluation.

## ION EXCHANGE CHARACTERISTICS OF EOR SYSTEMS

OAK RIDGE NATIONAL LABORATORY

DOE - \$100,000

4/1/77 - Continuing

**OBJECTIVES** — This research program seeks to clarify the complex interactions that occur between the micellar floods and oil-bearing formations over a wide range of salinities and alkali-to-alkaline earth ratios. The loss of surfactants on geological formations and their interactions with these solids are largely determined by the ion-exchange characteristics of both and by salting-in the salting-out effects. These effects are strongly dependent on the ion content of the solutions. Four major areas of research can be identified: ion-exchange equilibria between formations and contacting brines; effect of brine composition on phase behavior of aqueous-cosurfactant-surfactant-hydrocarbon systems; adsorption of cosurfactants and surfactants on minerals typical of formations; and the effect of characteristic materials, particularly clays, on adsorption of surfactants. The information elucidated may provide a basis for prediction of optimal compositions for injections into petroleum-bearing formations and of behavior in passage through the reservoir.

**RECENT WORK AND ACCOMPLISHMENTS** — Measurements of the distribution of Ca (II) and Na (I) on montmorillonite are in progress. Some surprising effects of the loading of the clay by the divalent ion on distribution coefficients leads to questions on present experimental procedures; data are being reviewed.

Investigation of phase relationships of simple systems, chemically similar to micellar flood compositions, continues. The miscibility region of alcohol-hydrocarbon-aqueous alkyl aromatic sulfonate solutions increases with increasing alkyl substitution on the benzene ring, even though the alkyl side chains on the sulfonates are well below the length thought to cause the formation of micelles. For a given aromatic sulfonate, the miscibility region decreases as the chain length of the hydrocarbons is changed in measurements covering the range from octane to pentadecane. Likewise, the amount of n-butanol required to produce miscibility with alkyl aromatic hydrocarbons increases with the length of the alkyl chain.



Measurements were made of the amounts of various alcohols (isomers of propyl and butyl alcohol) necessary to make miscible approximately 1:1 mixtures of hydrocarbon-aqueous salt solution systems. The hydrocarbons used so far have been toluene, cyclohexane, and decane with aqueous sodium p-toluene sulfonate solutions. The amount of alcohol required is larger for lower surfactant concentrations. In other experiments, the amount of alcohol required for miscibility was determined as a function of a series of various sulfonate surfactants.

**PLANS FOR THE COMING YEAR** – Measurements of alkali/alkaline earth equilibria will be extended to solids other than montmorillonite, including carbonates and other non-clays. Effects of temperature on phase equilibria will be determined. Phase studies with organic compounds will be continued and extended to include examples with alkyl side chains long enough for micelle formation, i.e., into the surfactant range. Anionic, neutral, and cationic surfactants may also be investigated. Distribution coefficients of ions between aqueous and oil phases will be determined. A study of adsorption of surfactants and cosurfactants on clays will be initiated.

## SELECTION OF MULTIPLE TRACERS FOR EOR SYSTEMS

OAK RIDGE NATIONAL LABORATORY  
DOE - \$100,000  
4/1/77 - Continuing

**OBJECTIVES** – Through a systematic search of the periodic table, this research program will identify those elements and ions that can serve as tracers for water flow under the conditions of salinity and alkali-alkaline earths ratios encountered in natural formations or at interfaces with floods.

Before an expensive chemical flood is undertaken the flow pattern of the formation must be established to assure that there are no serious losses or bypasses through fractures or “permeability streaks.” If they are not present, one can use water tracing to establish the volume of the formation that is to be processed by an injected fluid or “flood.” It is obviously of great importance to keep the volume of injected fluid as small as practical. Accurate knowledge of the volume of the formation to be treated is required to prevent use of excessive quantities of these expensive reagents and to evaluate the performance of the floods.

**RECENT WORK AND ACCOMPLISHMENTS** – For a compound or ion to be a good water tracer, conditions have to be identified under which adsorption is essentially negligible, which would probably mean low or negligible distribution coefficients computed per unit adsorptive capacity rather than per unit weight since low-capacity constituents often occur in truly enormous quantities. The search for ideal tracers cannot be separated from the liquid medium in which they are to operate. A species might be a suitable tracer in low salinity waters but not at high salinities and vice versa. Thus, it is essential to establish the adsorbabilities over a wide range of compositional variables from relatively dilute salt solutions to essential saturation with respect to salt and over broad ranges of monovalent-to-divalent counter ion ratios. A good water tracer also should not be significantly soluble in the oil phase of the formations; therefore, it will be necessary to determine for favored candidates distribution coefficients (extractabilities) by typical organic materials. Tracers favored at present include tritium-tagged water, several radioactivity simple anions, and several neutral organic compounds. The number available is not adequate to meet needs.

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Samples of montmorillonite from four sources were obtained and tested for selection and use in equilibrium studies. Distribution coefficients of  $\text{Cs}^+$ ,  $\text{K}^+$ , and  $\text{Ca}^{++}$  ions from concentrated sodium chloride solutions are being measured as a function of ionic strength using shaking experiments with montmorillonite as the adsorbent. Centrifuge and column techniques are also being employed. The development of techniques for rapid measurements continues to receive emphasis, as well as the acquisition of preliminary values of the distribution coefficients. Column measurements with swellable clays are difficult, although some encouraging results have been obtained with mixtures of clay with silica in various forms; for example, a column containing 80 percent Celite, a commercial filteraid, and 20 percent montmorillonite operated fairly well at 60°C with gravity flow. Although elution peaks were not completely symmetrical, distribution coefficients for the Cs (I)/Na (I) exchange were comparable to results from batch shaking experiments. Evaluation of the axial filter approach is also underway. In this mode, a feed containing the ion to be absorbed is passed through a chamber containing a slurry of a known amount of the absorbent. Effluent is removed through a filter mounted on a rotor in the chamber. The rotation hinders the buildup of a flux-limiting filter cake on the filter and stirs the suspension. The amount absorbed when effluent concentration equals feed concentration can be estimated by material balance and the distribution coefficients can then be computed. Alternatively the distribution coefficients can be obtained from the transient approach to steady state.

**PLANS FOR THE COMING YEAR** – The experimental conditions of the distribution coefficient measurements of tracers will be extended to salt solutions of anions other than chloride and to solids other than montmorillonite, such as kaolin, hydrous oxides, quartz, and carbonates. The effect of Na (I)/Ca (II) will be emphasized, and solutions will include divalent anions. Measurements of the distribution of tracers between aqueous and organic phases will begin.

## ENHANCED RECOVERY WITH MOBILITY AND REACTIVE TENSION AGENT

LAWRENCE BERKELEY LABORATORY  
DOE - \$130,000  
1977 - Continuing

**OBJECTIVES** – This present study will ascertain the tertiary-mode efficiencies of flooding acidic California crude oils with dilute aqueous bases, and devise laboratory screening tests that elucidate the governing recovery mechanisms and permit development of an improved mobility-reactive tension agent flooding package. The project involves studies on recovery mechanisms, displacement theory, interfacial tensions and charges, spontaneous emulsification, emulsion stability and emulsion rheology.

**RECENT WORK AND ACCOMPLISHMENTS** – A constant-rate, linear-displacement apparatus has been constructed that permits monitoring of pressure drop, electrical resistivity, streaming potential, and pH in addition to flowing oil and water fractions in cores up to 3 ft in length over a wide range of flow rates. The entire apparatus is thermostated in an air bath, and provision has been made to permit testing under anaerobic conditions so that field conditions can be simulated. Initial studies have begun with model systems of mineral oils, alkyl-aryl and alkyl carboxylic acids, caustic, and Ottawa sand. Work in this laboratory has indicated that tertiary oil can be recovered at low capillary numbers with well-defined systems that exhibit spontaneous emulsification. Oil production is in the emulsified state and is accompanied by large permeability reductions. This initial positive work reveals the importance of emulsions in high-pH flooding.

**PLANS FOR THE COMING YEAR** – Dynamic displacements with well-characterized chemical systems will be continued including the effect and scaling of linear flooding rate and the role of the porous medium morphology and surface chemistry. Dynamic pressure drops, electrical resistivity, streaming potentials, and flow visualization will aid interpretation of the reactive tension agent flooding mechanisms. Recovery efficiencies of promising California crude-oil-connate-water-preserved-core systems are being examined under an anaerobic environment and at elevated reservoir temperatures. Rheology and stability of high-pH emulsions in porous media are being studied to establish their mobility control and conformance characteristics.

## **LOW INTERFACIAL TENSION AND MISCIBILITY STUDIES FOR SURFACTANT PROCESSES**

CARNEGIE-MELLON UNIVERSITY  
DOE - \$168,360; Carnegie-Mellon University - \$8,862  
12/1/76 - 11/30/78

**OBJECTIVES** – A study of the role played by physical structures in surfactant solutions is being made. The morphology of the structures is to be examined under the polarizing microscope. The contribution of micelles to the interfacial properties will be determined by theoretical and experimental methods. Interfacial tension measurements on well-defined surfactants will be used to infer the properties of two-dimensional structures at the interface. It is hoped to achieve an understanding of the physical causes of low interfacial tensions, which would greatly simplify the process of choosing surfactant mixtures for chemical flooding.

**RECENT WORK AND ACCOMPLISHMENTS** – A correlation was found between birefringent phase changes in surfactant solutions and their interfacial behavior. The locus of salinities and surfactant concentrations for low interfacial tension and third-phase formation with oil is also the boundary between the one-phase region (lamellar) and the two-phase region (dispersion of liquid crystals). The theory of micelles and low interfacial tension was confirmed by measurements of micellar size in the ultracentrifuge. It was also found that in the salinity region where there was a third phase, this phase was water external (determined by direction of migration) at low salinity and oil external at high salinity, probably with an intermediate bicontinuous structure.

The “first CMC” for a surfactant is a critical phenomenon in the bulk phase. Surface tensions of sodium dodecyl sulfate showed a phenomenon called the “second CMC,” which is believed to be a critical phenomenon at the surface.

**PLANS FOR THE COMING YEAR** – The study of birefringent structures, both in bulk and at the

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## RESEARCH ON CHEMICAL OIL RECOVERY SYSTEMS

UNIVERSITY OF FLORIDA  
DOE - \$370,000  
12/76 - 5/78

**OBJECTIVES** – This program seeks to determine the various interactions occurring between injection fluids, such as surfactant and polymer solutions, and reservoir components, such as clays, minerals, oils, and sand. The research program is designed to establish a broad framework of information relating microstructure of injection fluids and their behavior in porous media to oil displacement efficiency. Development of the basic framework of new information and the quantitative relationships among various parameters of the process will assist industry in developing, optimizing, and fine tuning of injection fluids for varying reservoir conditions.

**RECENT WORK AND ACCOMPLISHMENTS** – An explanation of the molecular mechanism for achieving ultralow interfacial tension has been developed as part of the work on interfacial phenomena. The effect of surfactant concentration, salinity, alcohol background, and chain length of oil on partitioning and salt tolerance has been examined.

Viscosity studies in the area of bulk, surface, and porous media rheology indicated that brine solubility for alcohols plays a predominant role in influencing the micellar structure as compared to their molecular areas. The studies of polymer rheology have shown that capillary viscometry measurements are more sensitive to salt addition than either screen factor or extensional viscosity.

**PLANS FOR THE COMING YEAR** – Experimental work efficiency in sandpacks with different amounts of clays will be determined. Interfacial tension and surface viscosity measurements of oil/surfactant mixtures in the presence of dissolved polymers will be made.

## INTERFACIAL EFFECTS IN RECOVERY OF RESIDUAL OIL BY DISPLACEMENT

UNIVERSITY OF HOUSTON  
DOE - \$147,282  
4/1/77 - 3/31/79

**OBJECTIVES** – The role of dynamic interfacial properties (e.g., surface viscosities and elasticity) in the displacement of residual oil is not well understood. Such effects may play important roles in low-interfacial tension flooding. Proper evaluation of the effect is limited by inadequate techniques for the measurement of dynamic interfacial properties and the lack of accompanying displacement tests and analyses. The objective of this study is to develop and evaluate methods for the measurement of dynamic interfacial properties at oil-water interfaces, and to carry out displacement tests.

**RECENT WORK AND ACCOMPLISHMENTS** – Drop deformation, orientation, and circulation tests in simple shear field were performed on two liquid-liquid systems. For each of these tests, the surface viscosities of a given system agree within experimental errors; however, it appears only in the case of low-interfacial tension systems that the drop deformation-orientation tests provide measurement of the intrinsic surface shear and dilatational viscosities, and that the interfacial gradient effects play secondary roles. This finding is in agreement with prediction from theoretical

analysis, which concludes that the effect of mass transfer on drop deformation will complicate the measurement for high tension systems.

A deep-channel surface viscometer has been constructed, and tested on a lauryl sulfate-air system. It was shown that impurities' presence in commercial lauryl sulfate creates an anomalous aging effect on the surface viscosities' measurement. An experimental system for studying oil globule displacement dynamics in microcapillaries has been designed and is under construction. A porous media model for studying oil ganglia motion, entrapment, and coalescence has been formulated. A computer algorithm for simulating such ganglia motion has been developed. The results of some preliminary calculations indicate that this model can predict accurately the relative permeability to water in an oil-water-sandstone system. Only basic data of the system are needed for the calculations; no curve fitting is required.

An analysis of the efficiency of a flooding process without ganglia coalescence has been completed. The results showed that this flooding process is very inefficient and serves to underline the crucial importance of coalescence to successful chemical flooding; therefore, a model to simulate ganglia coalescence and breakup was proposed. Two coupled ganglia-number conservation equations, one applied to moving ganglia and the other to a stranded one, were developed. Further analysis on this problem is in progress.

**PLANS FOR THE COMING YEAR** – Deformation, orientation, and circulation experiments will be performed on a number of fluid systems. The measured surface viscosities will be verified using the deep channel surface viscometer. Analysis of ganglia motion in the porous media model will be continued taking into account the ganglia coalescence and breakup phenomena. Experimental studies of ganglia displacement in microcapillaries and micromodel will be initiated.

## MECHANISM OF OIL BANK FORMATION AND COALESCENCE IN POROUS MEDIA

ILLINOIS INSTITUTE OF TECHNOLOGY

DOE - \$452,000

9/1/76 - 9/1/77

**OBJECTIVES** – A basic study of the rupture and coalescence of oil droplets in water is being made and how it depends on interfacial rheology will be established. It is planned to make observations under a variety of conditions, such as changes in salinity, and in particular to compare behavior in surfactant solutions with that in caustic. The results will be correlated with oil recovery by a study of the formation of the oil bank in chemical flooding.

**RECENT WORK AND ACCOMPLISHMENTS** – Techniques were developed for observing the coalescence of emulsions, and also of individual droplets with interfaces and with each other. Very reproducible coalescence times were obtained in an apparatus allowing close control of drop size. Coalescence rate was maximum for systems that formed a third phase, especially if the third phase was a clear microemulsion. A cinemicrophotographic technique was used to observe coalescence and spontaneous emulsification in micromodels of porous media. The appearance was quite different in surfactant and caustic solutions. Films that form around the drops were found to have an important influence on coalescence, and also on oil recovery. Good recovery was associated with thin but finite films (0.5 micron), and with rapid coalescence.

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Methods were developed for measuring a complete suite of interfacial rheological properties including viscosity and elasticity, each of which may be associated with shear or dilational stresses, and the origin may be either intrinsic or "compositional" (i.e., dependent on mass transfer and determined by calculation from diffusion, etc.). Several well-defined surfactant systems and a polymer solution were measured and found to exhibit varying relations between viscosity and elasticity, and between intrinsic and compositional components. No pure system has been found with low interfacial tension and positive interfacial viscosity. Only crude oil showed a measurable surface viscosity. The conclusion can be drawn that modeling crude oils with alkanes is inadequate.

**PLANS FOR THE COMING YEAR** – The methods developed will be applied to a study of the interfacial properties and their correlation with coalescence over a variety of physicochemical conditions. A quantitative comparison of surfactant and caustic behavior will be made. The nature of the interfacial films will be investigated to identify them as adsorbed layers or incipient formation of a third phase. The cinemicrophotographic technique on micromodels will be exploited to study phenomena of oil bank formation and recovery.

**ULTRALOW INTERFACIAL TENSION, PHASE BEHAVIOR,  
AND CHEMICAL FLOODING PROCESS**

UNIVERSITY OF MINNESOTA  
DOE - \$163,500  
12/77 - 9/78

**OBJECTIVES** – This program seeks to determine answers at a fundamental level to the questions: How do effective surfactant molecules produce tensions less than 0.01 dyne/cm at oil-water interfaces? How is residual oil mobilized and kept moving in chemical flooding processes? and How best can oil displacement in chemical flooding processes be simulated for purposes of design and optimization? Results will elucidate the mechanisms by which surfactants behave and will give valuable information on displacement of one fluid by another in porous media. These explanations will be a major contribution to basic knowledge relating to the long-range problem of full utilization of petroleum resources.

**RECENT WORK AND ACCOMPLISHMENTS** – An explanation of the relation of interfacial tension to intermolecular forces has been developed. Experimental work has confirmed two regimes of low interfacial tension, one associated with microemulsion phases at equilibrium, the other with oil-water interfaces probably not in two-phase equilibrium. Studies have been made on order-of-mixing effects and slow equilibration phenomena for surfactant systems. A thermodynamic model has been developed that represents the essential aspects of surfactant-brine-oil phase behavior including metastable equilibria. Conceptual identification has been made of the possibility of bicontinuous structures in microemulsion phases. Freeze etching and electron microscopy have shown that middle-phase microemulsions in certain commercial petroleum sulfonate systems are neither globular, tubular, nor lamellar in microstructure. A short motion picture film with soundtrack has been produced that show a single residual oil blob in a scaled-up model of a porous media is mobilized at about the capillary number expected from literature correlations of microscopic displacement efficiency, and the ideas of competition between capillary pressure difference and Darcy pressure difference. A theoretical explanation in terms of percolation concepts and topology of pore space has been made of accessible porosity, conductivity, relative

permeability, and capillary invasion. It has been theoretically shown that if phase behavior of a surfactant system is sufficiently favorable, good oil recovery can be attained without low interfacial tension.

**PLANS FOR THE COMING YEAR** – Further modifications of the spinning-drop apparatus are planned. Instrumentation and techniques will be developed for electron microscopy studies of interfaces and microemulsions. The statistical thermodynamics of microemulsions and microemulsion tensions will be developed. Phase behavior and colloidal dispersions will be studied using the ultracentrifuge. Mathematical simulation of pseudoternary and pseudoquaternary phase behavior will be made. The percolation theory of dispersion including mass transfer effects will be extended. Scaled-up experiments on blob mechanics are planned and will be related to percolation concepts including the theory of permeability. The effect of reservoir heterogeneities on permeabilities and displacement will be explored.

## INTERFACIAL EFFECTS IN RECOVERY OF RESIDUAL OIL BY DISPLACEMENT

NORTHWESTERN UNIVERSITY

DOE - \$181,959

5/1/77 - 4/30/79

**OBJECTIVES** – Preliminary analysis and initial laboratory data indicate the influence of dynamic interfacial properties (interfacial viscosity, contact angle hysteresis, and wettability) on tertiary oil recovery efficiency. These claims, if validated, will greatly change the direction of research in this area. The objectives of this work are to analyze in detail the displacement process during oil recovery and to provide reliable data for such an analysis.

**RECENT WORK AND ACCOMPLISHMENTS** – A model for the displacement of oil in porous media was set up, and a computer program for this analysis, based on iterative procedure, has been completed. A test run was made on a simplified model of a liquid displacing a gas in a straight capillary in a Poiseuille flow. The model was capable of predicting the velocity gradient of such a flow correctly. An alternative method of solving this problem, which is based on computing the upper and lower bound for the rate of dissipation of energy within the system, has also been developed. An analysis has been made on the spinning drop method of measuring surface viscosity. The feasibility of designing an experiment based on this method is under study.

The meniscal breakoff equipment was modified using pressure transducers to indicate the breakoff point. This method is precise, accurate, and required much less time to reach equilibrium than the widely used spinning drop method. This work also raised the question as to whether the interfacial tension data reported in the literature are true equilibrium values.

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## IMPROVED POLYMERS FOR EOR SYNTHESIS AND RHEOLOGY

UNIVERSITY OF SOUTHERN MISSISSIPPI  
DOE - \$336,729; University of Southern Mississippi - \$70,744  
9/15/77 - 9/30/80

**OBJECTIVES** – Commercial polymers, properly formulated, have shown great potential as oil displacement fluids in EOR. Unfortunately, a number of problems including thermal and mechanical degradation, adsorption, chromatographic effects, shear thinning, electrolyte effects, and structural inhomogeneities have limited the effectiveness of these systems in field tests. This research program will synthesize and investigate the rheological properties of water-soluble copolymer systems to function as mobility control agents in EOR. The data from these studies will be utilized to improve the flow properties of polymer solutions through porous media under laboratory and simulated field conditions. A further goal of the research will be to develop quick laboratory screening methods for potential EOR polymers. The final goal is the education and training of young scientists for future energy R&D activities.

**RECENT WORK AND ACCOMPLISHMENTS** – This is a new project, and the selection of capable graduate students to work with the principal investigator in the various areas of the proposed research program has been completed. The proposed research is based on extensive studies previously made in the University's polymer laboratories of existing commercial systems and promising new synthetic graft copolymers.

**PLANS FOR THE COMING YEAR** – A systematic attack on a number of these problems at a fundamental level will be made beginning with macromolecular structure and proceeding to macroscopic flow properties. The research will be conducted under a coherent, integrated program involving: polymer synthesis; characterization; and solution rheology including physics of polymer fluid flow through porous media. Controlled experiments are designed to solve some of the complex problems associated with chemical and physical polymer interactions under expected operational conditions including temperature, pressure, time, flow rate, and ionic strength. Tasks for the coming year will be the characterization of existing commercial polymer molecules and the determination of the rheological properties of aqueous solutions containing such polymers. In addition, the synthesis and characterization of new polymers will also be done.

## SPONTANEOUS EMULSIFICATION AS A TERTIARY OIL-RECOVERY MECHANISM

UNIVERSITY OF TEXAS AT AUSTIN  
DOE - \$62,966  
8/1/75 - 7/31/77

**OBJECTIVES** – This project was designed to examine the extent to which residual oil can be mobilized by spontaneous emulsification. The results could indicate whether this mechanism merely introduces errors into laboratory flood tests, or whether it also has potential as a recovery technique in the field. If so, it could be less sensitive to changes in salinity or surfactant concentration than the low-tension processes.

**RECENT WORK AND ACCOMPLISHMENTS** – The model for the diffusion and stranding mechanism of spontaneous emulsification was tested experimentally. It was verified that phase



boundaries move at a rate proportional to the square root of time. The model was extended to the systems that form three phases, which are considered optimal in current technology. No way was found theoretically or experimentally to distinguish between multiple mathematical solutions; however, the region of multiple solutions on the ternary phase diagram was delineated. The upper boundary of the single-solution region is formed by drawing a line from the initial composition point through the plait point to the side of the diagram. When this line becomes tangent to the curve between two-phase and one-phase regions, the two-solution region shrinks to zero.

**PLANS FOR THE COMING YEAR** – This project will be merged into the larger project, Tertiary Oil Recovery Processes, supported by DOE and industrial grants. Efforts will be directed toward including previously ignored ternary diffusion effects. Also, experiments will be designed to distinguish spontaneous emulsification from oil swelling, both of which are predicted for the same systems.

### ENHANCED OIL RECOVERY

#### MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$420,000

7/67 - Continuing

**OBJECTIVES** – Methods of increasing the recovery of crude oil from pressure-depleted low-productivity reservoirs are being evaluated with field and laboratory tests. There is evidence that carbon dioxide and micellar fluid injections have increased the producing life of certain oil reservoirs. This study is designed to provide data relative to the extent of control that can be exercised for secondary and tertiary recovery in characterized formations. These R&D activities will be expanded to western carbonate oil reservoirs as determined by the EOR management plan.

**RECENT WORK AND ACCOMPLISHMENTS** – The following are the studies continuing or developed during FY 77:

| Oil Field      | Contractor                   | Location          | Slug Type       | Date        | Lab Study   |
|----------------|------------------------------|-------------------|-----------------|-------------|-------------|
| Granny Creek   | Columbia Gas                 | Clay County WV    | CO <sub>2</sub> | 1/76-cont.  |             |
| Hilly Upland   | Allegheny Land & Mineral Co. | Lewis County WV   | CO <sub>2</sub> | 10/76-12/77 |             |
| Rock Creek     | Penzoil Co.                  | Roane County WV   | CO <sub>2</sub> | 2/76-cont.  |             |
| Weeks Island   | Shell Oil Co.                | Iberia Parish     | CO <sub>2</sub> | 6/76-cont.  |             |
| Griffithsville | Guyan Oil Co.                | Lincoln County WV | CO <sub>2</sub> | 8/75-8/78   |             |
| Bradford       | Penn-Grade Assoc.            | Bradford, PA      | Micellar        | 9/75-cont.  |             |
| Bradford       | PA S.U.                      | Univ. Park        | Micellar        | 8/75-cont.  | Micellar    |
| Bradford       | MERC                         | MERC              | Sulfonates      | 4/77-6/78   | Sulfonates  |
| Hilly Upland   | MERC                         | MERC 7BI-I        | CO <sub>2</sub> | 4/77-2/78   | Model Study |

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Approximately 9000 barrels of tertiary oil have been recovered from the *Columbia Gas* CO<sub>2</sub> injection project. Production from the pilot producer has declined to less than 3 bbl/d, and the water/oil ratio is nearly 10:1. *Pennzoil Company* has injected over 650,000 barrels of water into their CO<sub>2</sub> injection project. Injection rate into 19 wells is 2950 bbl/d at 750 psi wellhead pressure. *Shell Oil Company's* CO<sub>2</sub> injection project in the Weeks Island field in Louisiana is just underway. Preliminary design work is nearly complete and a contract has been let for drilling the production well. Reclamation of well sites and roads is essentially complete on *Guyan Oil Company's* CO<sub>2</sub> injection project. Reservoir pressurization and field testing has been started. Initial water/oil ratios from this "dump-flooded" area range from 1:1 to over 10:1. Micellar slug design, injection plans, and facilities are essentially complete for the *Penn-Grade Crude Oil Association's* micellar-polymer project in the Bradford field. Micellar slug injection was initiated September 12, 1977.

A 1500-ton injection slug of CO<sub>2</sub> was completed, and water injection was initiated into the Hilly Upland Oilfield. One of two surrounding wells is responding to the injection with increased oil production. Several tertiary oil recovery runs made with linear Berea cores indicate the possibility (contrary to conventional thinking of the surfactant flooding industry) that water-soluble petroleum sulfonate fractions of molecular weights in the thousands and minor amounts of polyacrylamides can recover far more tertiary oil per unit weight of chemical than current micellar floods.

**PLANS FOR THE COMING YEAR** — The following projects are scheduled for completion: technical implementation plan on field testing in carbonate oil reservoirs by CO<sub>2</sub> injection; investigation of EOR mechanisms using water-soluble sulfonated hydrocarbons; CO<sub>2</sub> injection test in Hilly Upland reservoir and the associated laboratory displacement studies; investigation of mechanisms, displacement, and phase behavior using CO<sub>2</sub> injection; and mathematical modeling techniques for predicting results of CO<sub>2</sub> flooding for enhanced recovery. The western field testing projects will be initiated during the year, and evaluation of the Hilly Upland reservoir injection test and studies will be made.

## OIL RECOVERY BY CARBON DIOXIDE INJECTION

COLUMBIA GAS TRANSMISSION CORPORATION  
DOE - \$472,000; Columbia Gas - \$944,000  
6/1/76 - 6/1/79

**OBJECTIVES** — This project is designed to demonstrate the efficiency and economics of recovery oil from a shallow low-temperature, watered-out reservoir using carbon dioxide and water to displace the oil for tertiary recovery. This method of enhanced recovery may be capable of producing substantial quantities of the remaining residual oil. Under certain conditions, carbon dioxide is miscible with crude oil and, as in any form of miscible displacement, up to 100 percent of the oil contacted by CO<sub>2</sub> under conditions of miscibility can be recovered.

**RECENT WORK AND ACCOMPLISHMENTS** — Carbon dioxide injection was completed, June 14, 1977, with a total of 9878 tons injected into the four injection wells. Water injection was then resumed to drive the CO<sub>2</sub> slug through to the producing well. Water injection rates average 45 bbl/d/well. Over 8000 barrels of additional oil production has been attributed to CO<sub>2</sub> injection, with over 3500 barrels from the center production well. Bottom-hole pressures within the pilot ranged from 1800 psi at the injection wells to approximately 900 psi at the center production well. This well was put on pump during August to increase fluid production from within the pattern. By

September the water production had increased considerably with a daily production of 2 to 4 barrels of oil and 20 to 25 barrels of water.

**PLANS FOR THE COMING YEAR** – Water injection to chase the CO<sub>2</sub> slug through the reservoir will continue. Fluid and gas production will be monitored very closely to determine changes in percent of CO<sub>2</sub> produced and percent of water and oil produced. Technical evaluation of the project will also continue. To date, the results of this project are encouraging.

## OIL RECOVERY BY CARBON DIOXIDE INJECTION

PENNZOIL COMPANY  
DOE – \$1,086,750; Pennzoil - \$1,579,550  
7/1/76 - 7/1/81

**OBJECTIVES** – This project is designed to demonstrate the efficiency and economics of recovering oil from a shallow low-temperature reservoir using carbon dioxide and water to displace the oil for secondary recovery. The primary mechanism responsible for the increased recovery will be a multiple contact miscible drive. The success of this project will result in part from swelling of the residual oil, reduced oil viscosity, and lower interfacial tension.

**RECENT WORK AND ACCOMPLISHMENTS** – This project is located in the Rock Creek Field, West Virginia. During the year, 664,000 barrels of water have been injected into 19 injection wells at an average wellhead injection pressure of 790 psig. Fluid and gas production has been monitored for the two center production wells to assure good production data. An analysis of the reservoir core data for the pilot area shows the effective sand thickness to be 32.4 feet with an average porosity of 21.9 percent. Average permeability was determined to be 20.5 millidarcys; oil saturation was calculated to be approximately 34 to 38 percent of pore volume.

A new water supply facility, located 5 miles south of the project, was constructed. The old source had a high iron content and developed problems with treatment. The new supply will provide approximately 3000 bbl/d of water for injection. The CO<sub>2</sub> storage (4 tanks capable of holding 44 tons) and injection facilities were installed and are now ready.

**PLANS FOR THE COMING YEAR** – Reservoir pressures will be determined by pressure falloff tests on several injection wells. When it has been increased to approximately 1000 psi, CO<sub>2</sub> injection will be started (planned for early to mid-1978), with 15,000 tons to be injected by the WAG method over a 6-to-9-month period.

## OIL RECOVERY BY CARBON DIOXIDE INJECTION

SHELL OIL COMPANY  
DOE - \$2,475,000; Shell - \$3,025,000  
6/10/77 - 3/1/81

**OBJECTIVES** – This project seeks to demonstrate that carbon dioxide miscible displacement can be successfully accomplished in deep, hot reservoirs, which are unsuitable for surfactant flooding (a unique application of CO<sub>2</sub> injection in tertiary oil production projects); to provide incentive for

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development of other CO<sub>2</sub> projects along the Gulf Coast and for the development of adequate CO<sub>2</sub> supplies); and to transfer technology developed to private companies for further commercial application in other similar situations along the Gulf Coast.

**RECENT WORK AND ACCOMPLISHMENTS** — This project is located in the Weeks Island Field, Iberia Parish, Louisiana, and is a tertiary recovery project as the reservoir has been waterflooded. The field is a piercement-type salt dome and the reservoir occurs at a depth of 12,800 feet, with a temperature of 225°F and a bottomhole pressure of 5100 psi. The reservoir dips at 27 to 52 degrees. Porosity of the reservoir has been determined to average 23 percent and contain a residual oil saturation of 27 to 37 percent. Experiments are being made to determine percent of methane to be added to the CO<sub>2</sub> slug for a gravity stable miscible displacement. Also mathematical modeling of the reservoir was started. Plans were completed to drill, core, log, and test a new oil production well for the project. Also, plans were completed for CO<sub>2</sub> storage and injection facilities for the project.

**PLANS FOR THE COMING YEAR** — The oil production well will be drilled, cored, and tested to determine the reservoir characteristics such as porosity, permeability, and oil saturation. The experiments discussed above will be completed. Injection of the CO<sub>2</sub> slug will start and continue until 570,000 reservoir barrels of the CO<sub>2</sub> methane mixture is injected; 50,000 tons of CO<sub>2</sub> will be used over a 12-month injection period. Concurrently, salt water production will commence from a downdip converted water injection well at a rate of 1500 bbl/d of salt water. Water will be produced from perforations at approximately 13,230 feet. All phases of the work will be closely monitored to provide data for process evaluation.

## CARBON DIOXIDE RECOVERY AND TERTIARY OIL PRODUCTION ENHANCEMENT

AMERON  
DOE - \$150,951  
9/12/77 - 9/11/78

**OBJECTIVES** — This study will locate commercial sources of carbon dioxide in the Los Angeles Basin, determine which local oil reservoirs are amenable to CO<sub>2</sub> enhanced oil recovery, and list the requirements for treating and transporting CO<sub>2</sub> gas to those potentially high candidate reservoirs for CO<sub>2</sub> flooding.

**RECENT WORK AND ACCOMPLISHMENTS** — This project has been initiated.

**PLANS FOR THE COMING YEAR** — A literature search will be conducted on sources of CO<sub>2</sub> (such as oil refineries, ammonia plants, electric power plants, etc.). Personnel of such plants in the Los Angeles Basin will be contacted to ascertain the quantity of available CO<sub>2</sub>. An optimal pipeline and compressor system will be proposed. Problems of corrosion and handling of CO<sub>2</sub> will be discussed with field experts. Material specifications for handling and treating the CO<sub>2</sub> will be presented. Another literature search will be made and experts consulted on ranking of candidate reservoirs for CO<sub>2</sub> enhanced oil recovery potential.

## ENHANCED OIL RECOVERY BY CARBON DIOXIDE INJECTION

PULLMAN KELLOGG COMPANY

DOE - \$136,909

9/27/76 - 9/1/77

**OBJECTIVES** – This study identified oil reservoirs/fields thought to be suitable for EOR by CO<sub>2</sub> miscible flooding, and estimated CO<sub>2</sub> requirements and potential oil recovery for these reservoirs/fields; surveyed available CO<sub>2</sub> supplies within these geographic areas; and developed systems to purify and deliver CO<sub>2</sub> (including associated costs) for several hypothetical cases including above-ground and natural sources.

**RECENT WORK AND ACCOMPLISHMENTS** – A screening procedure was used to locate candidate fields/reservoirs suitable for EOR by CO<sub>2</sub> miscible flooding. It was applied to a data bank of over 2500 reservoirs maintained by Gulf Universities Research Consortium. Only 1.5 to 2 percent of the fields meet the criteria. Estimates were made of potential oil recovery (3.2 trillion bbl) and CO<sub>2</sub> requirements (9.2 trillion scf) for the states with most promising candidate fields—Texas, Louisiana, Mississippi, and California. Above-ground sources of CO<sub>2</sub>, including power plant and cement plant stack gases and process plant vents, are estimated at 33 billion scf/d. Although this is sufficient to satisfy the projected demand for EOR applications, roughly 80 percent is presently available as a byproduct from cement plant or power plant stack gases and is of low quality. Recovery of CO<sub>2</sub> from these sources will be expensive because of high-separation costs which could prevent extensive use of these abundant sources. The highest quality above-ground sources of CO<sub>2</sub> are process vents from fertilizer and chemical plants. Presently, uncommitted byproduct CO<sub>2</sub> from ammonia manufacture (typically 98 percent CO<sub>2</sub>) is a most promising process vent source since about 70 percent is located in areas of high potential for EOR by CO<sub>2</sub> miscible flood—namely, Louisiana, South Texas, and Oklahoma. Byproduct CO<sub>2</sub> from SNG plants has even greater potential as a process vent source of CO<sub>2</sub> for EOR, but only if a significant amount of SNG is produced by gasification of coal. Natural gas wells rich in CO<sub>2</sub> have perhaps the greatest potential; however, the exact quantities available are not known. The Four Corners area (Utah, Colorado, Arizona, New Mexico), Southeast Colorado, Northeast New Mexico, and Central Mississippi are believed to be rich in high-purity naturally occurring CO<sub>2</sub> wells. Based on cost data developed during this study, the lowest cost CO<sub>2</sub> would be from a natural source containing sufficient salable natural gas to offset development expenses and delivering CO<sub>2</sub> to an EOR project. Realistically, it seems that such sources would most likely be developed independently of EOR efforts for the natural gas content alone.

**PLANS FOR THE COMING YEAR** – The second phase of this study will include locating all significant sources of CO<sub>2</sub> on regional maps in a quantitative manner so that a description in terms of quantity, quality, and initial conditions is available; evaluating all the costs associated with delivering CO<sub>2</sub> from its source to its destination; and making analyses for selected regions considered to be the best EOR prospects by CO<sub>2</sub> miscible flooding. A point within the region will be selected as the delivery point for CO<sub>2</sub> and the cost of delivering it will be determined.

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## STIMULATING HEAVY-OIL PRODUCTION

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$650,000

1973 - Continuing

**OBJECTIVES** – This project is aimed at developing economical and efficient methods for production from heavy-oil reservoirs, thus increasing available petroleum reserves at a minimum cost to the consumer without damaging the environment. Three recovery methods being investigated are explosive-fracturing; solvent-injection (SolFrac), in-situ combustion, and carbon dioxide injection. It is planned to pilot test these methods in a low-gravity (15 degrees API) crude oil reservoir.

**RECENT WORK AND ACCOMPLISHMENTS** – Results of the *SolFrac* field test have been documented and should be published soon. Laboratory testing is underway, which is to be followed by a field experiment to test the technical feasibility of recovery of crude oil by *in-situ combustion* from heavy oil reservoirs. Three in-house combustion tube tests were run using cores from the R.L. Link lease; two runs used cores having simulated fractures, and about 75 percent of the core was burned. The other test did not use fractured cores, and combustion was complete. The R.L. Link lease will be used for an in-situ combustion field test. This property is north of Bartlett, Kansas, and about ¼ mile southwest of the Leap lease on which the SolFrac experiment was performed. The first well was drilled and casing was cemented to a depth of 420 feet. Core and log preliminary indications showed several oil-saturated sand zones in the Bartlesville sand. Four additional wells were drilled in preparation for the field experiment. Air injection tests were performed after perforating the zone from 335 to 367 feet. The well would not take air at a maximum pressure of 340 psi. Subsequent attempts to inject air into the well were unsuccessful. The wells will be hydraulically fractured later. A trailer has been moved to the field site, electrical power installed, and other lease equipment and services obtained.

Laboratory studies are being made to test the feasibility of *CO<sub>2</sub> injection* for the recovery of crude oil from heavy-oil reservoirs that are shallow and contain no reservoir energy. All laboratory equipment items have been pressure tested and calibrated for use in the flow system, and permanently placed in their respective locations inside and outside the oven, etc. The system operates very satisfactorily at the pressures and temperatures for which it was designed; however, during the test runs, some trouble was experienced, the worst being failure of the flow pump seals at 800-psi pressure. Runs have been completed without complications at 200, 400, and 600 psi pressure levels at room temperature; the gear pump, used as a circulating pump, started leaking when the pressure level was raised to 800 psi. A centrifugal pump that can withstand high pressure on suction and discharge is being investigated. Results from the tests were as anticipated. Viscosities decreased from a high of 11,500 cp with no CO<sub>2</sub> at atmospheric conditions; density of the oil was decreased by the addition of CO<sub>2</sub>; and the CO<sub>2</sub>-oil ratio (cc CO<sub>2</sub> per cc oil) increased from 13.2 at 200 psi to 43.7 at 600 psi.

**PLANS FOR THE COMING YEAR** – Equipment will be procured and installed, and a contract negotiated for ignition services. All wells will be hydraulically fractured and air injectivity tests run. Wells will be ignited and production monitored. Laboratory investigation of CO<sub>2</sub>-heavy oil recovery will be continued at pressures up to 5000 psi and temperatures to 200°F.

## TAR SAND OIL RECOVERY BY IN-SITU COMBUSTION

LARAMIE ENERGY RESEARCH CENTER

DOE - \$1,290,000

6/72 - Continuing

**OBJECTIVES** – This project is to demonstrate the technical feasibility of employing in-situ combustion processes for the recovery of oil from U.S. tar sands. The tar sand resource contains nearly 30 billion bbl of low-gravity and extremely high-viscosity oil, most of which is beneath overburden too thick for surface mining. Over 90 percent of this oil is contained in six giant deposits in Utah. In-situ thermal methods, including combustion, represent the most promising processes for commercialization of this resource.

**RECENT WORK AND ACCOMPLISHMENTS** – This project began in 1972 with establishment of a laboratory for analyses of the physical properties of tar sands. Nearly 8000 samples from 12 Utah tar sand deposits and 1 New Mexico deposit have been analyzed and reported. The second field experiment (LERC TS 2C) was ignited in late summer of 1977 and will be completed in early 1978. This experiment is utilizing the reverse combustion process to condition the reservoir for subsequent application of the forward combustion process. Technical success for this experiment was indicated when nearly 20 percent of the oil-in-place was produced at the approximate midpoint of the experiment. The first and second field experiments utilize identical 9-well line patterns. Each pattern includes a row of three air-injection wells on either side of a row of three ignition-production wells with 20 ft distance between like wells and 60 ft distance between rows (overall pattern dimensions 40 ft by 120 ft). Several instrumented monitor wells are located in and adjacent to each well pattern. Depths of the producing intervals for the first and second field experiments are 300 and 350 ft, respectively. Thicknesses of the producing intervals are 11 and 13 ft.

**PLANS FOR THE COMING YEAR** – The second in-situ combustion experiment will be completed and resultant data will be analyzed. A companion project to demonstrate the feasibility of the utilization of steam for in-situ extraction of oil from tar sands has been approved. This project, which is included in the Enhanced Oil Recovery Management Plan, will be initiated in early 1978.

## DEVELOPMENT AND FIELD TESTING OF VAPOR THERM PROCESS

CARMEL ENERGY INC.

DOE - \$740,971; Carmel Energy - \$164,513

4/1/76 - 9/30/77

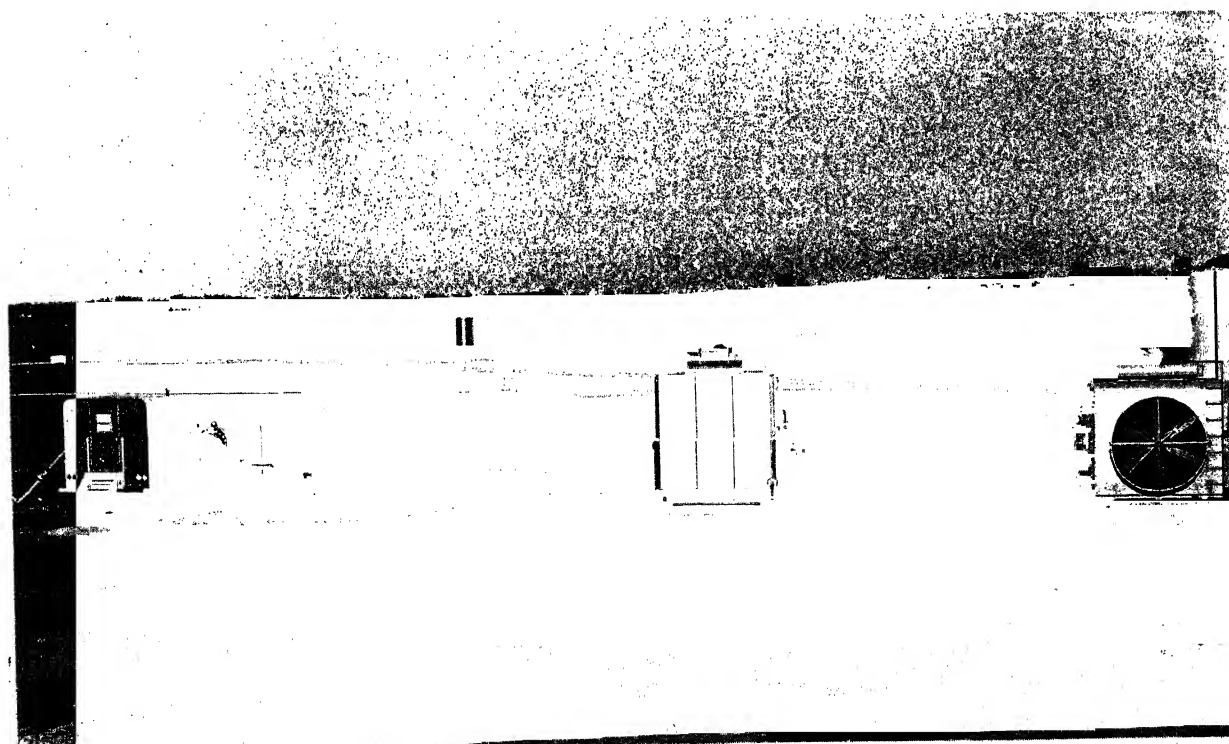
**OBJECTIVES** – The applicability of the Vapor Therm process to the extraction of high viscosity crude oil from the Bartlesville sand in southeastern Kansas is being determined. This process consists of injecting a mixture of steam and inert gases into the producing sand and then producing the oil from the same well using the cyclic stimulation technique. If successful, it could substantially increase the oil recovery from the many heavy-oil deposits and low-production reservoirs found in southeastern Kansas, northern Oklahoma, and western Missouri.

**RECENT WORK AND ACCOMPLISHMENTS** — Three injection-production cycles have been completed and a fourth has begun; the results are shown below:

| Cycle | (MMBtu) | Production Time (Days) | Oil Produced (Bbl) | Ratios        |                   |
|-------|---------|------------------------|--------------------|---------------|-------------------|
|       |         |                        |                    | MMBtu Bbl Oil | Bbl Oil Bbl Steam |
| 1     | 516     | 56                     | 1112               | 0.46          | 0.75              |
| 2     | 560     | 60                     | 1917               | 0.29          | 1.20              |
| 3     | 580     | 96                     | 3121               | 0.19          | 1.89              |
| 4     | 570     |                        |                    |               |                   |

During the production phase of the first cycle, difficulty was experienced in handling the produced fluids because emulsions were formed that increased the fluid viscosity by as much as 15 times that of the normal crude oil; however, in subsequent cycles, an emulsion destabilizer was added to the injection fluid, which solved this problem. A peak production of 114 bbl/d was reached in the second cycle and a peak of 63 bbl/d in the third. Significantly, however, after about 2 weeks both cycle production rates leveled off to a relatively constant 30 bbl/d of oil.

As a result of an economic evaluation of the Government-supported test, a commercial development has begun in the same field about a mile from the test site. Forty wells are being drilled on a 120-acre lease that will be stimulated by a new Vapor Therm Unit.



*Vapor Therm Unit Used To Stimulate Four Wells in Eastern Kansas with Its Vapor Therm Process (Shown on Left)*



**PLANS FOR THE COMING YEAR** – After obtaining data on the fourth production cycle, a final report will be submitted. Testing of the Vapor Therm process in a different field containing a lower degree API gravity oil will be performed.

### IMPROVED OIL RECOVERY BY STEAMFLOODING

CHANSLOR-WESTERN OIL AND DEVELOPMENT COMPANY  
DOE - \$1,700,000; CWOD - \$6,547,266  
6/76 - 6/83

**OBJECTIVES** – Cyclic stimulation has been the principal stimulation method of recovering oil from low-gravity heavy-oil reservoirs. Generally, successful cyclic steam projects are converted to steam-drive projects to accelerate production; however, numerous heavy-oil reservoirs have not responded to cyclic stimulation. A successful project would encourage the development of the drive process in other areas that were unresponsive to the cyclic process. Thus, the operational and economic aspects of steamflooding a flat low-pressure heavy-oil reservoir that has had an unfavorable response to cyclic steam injection are being demonstrated. Well pattern design and well completion technology are being evaluated.

**RECENT WORK AND ACCOMPLISHMENTS** – Approximately 500,000 bbl of steam were injected into four injection wells this year. Production wells were each stimulated with about 8000 bbl of steam to heat the area around the well bores, and steam injection profiles were obtained regularly. Temperature surveys were run every 6 weeks in the producing wells. An additional 22 million Btu steam generator was installed to increase the steam injection rate in the pilot area, which required installation of another water supply line. Two additional wells were drilled and placed on production offsetting the pilot area to the east and south. These wells will provide geological data for use in studies regarding expansion of the project area.

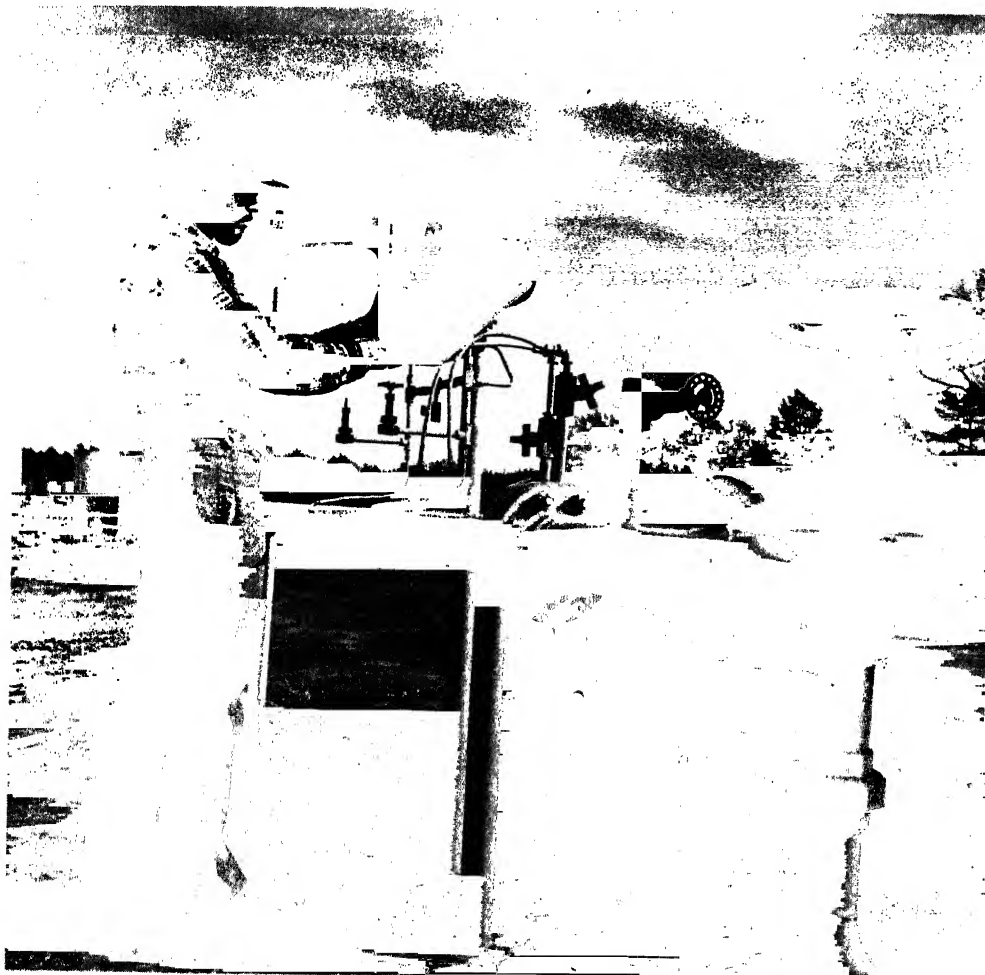
**PLANS FOR THE COMING YEAR** – The steam drive will be continued in the pilot area. To evaluate the project, steam profiles and temperatures surveys will be obtained and two wells will be drilled. Expansion of the project area will be initiated if the evaluation of the pilot project warrants it.

### IMPROVED OIL RECOVERY BY IN-SITU COMBUSTION

CITIES SERVICE COMPANY  
DOE - \$3,102,000; Cities Service - \$5,127,000  
6/30/76 - 6/30/82

**OBJECTIVES** – This project is being conducted in the Nacatoch Sand of the Bellevue Oil Field in northern Louisiana. Because of very low reservoir pressure and high oil viscosity, primary production rates are too low for economic operation; however, pilot firefloods in the Bellevue Field have been technically successful and indicate that combustion can economically recover a large fraction of the oil in place. The demonstration is expected to yield 700,000 bbl of oil during the life of the project, and as much as 30 million bbl may be recovered by fireflood from adjacent acreage. Successful demonstration of the combustion process and the operational techniques required could encourage exploitation of many other reservoirs. This project will investigate, by a large-scale field operation, the efficiency and economics of a simultaneous water-air-in-situ combustion process (fireflood), and demonstrate pilot-tested techniques of increasing vertical sweep efficiency (or process coverage). Process design will emphasize reduction of project time to maximize revenue.

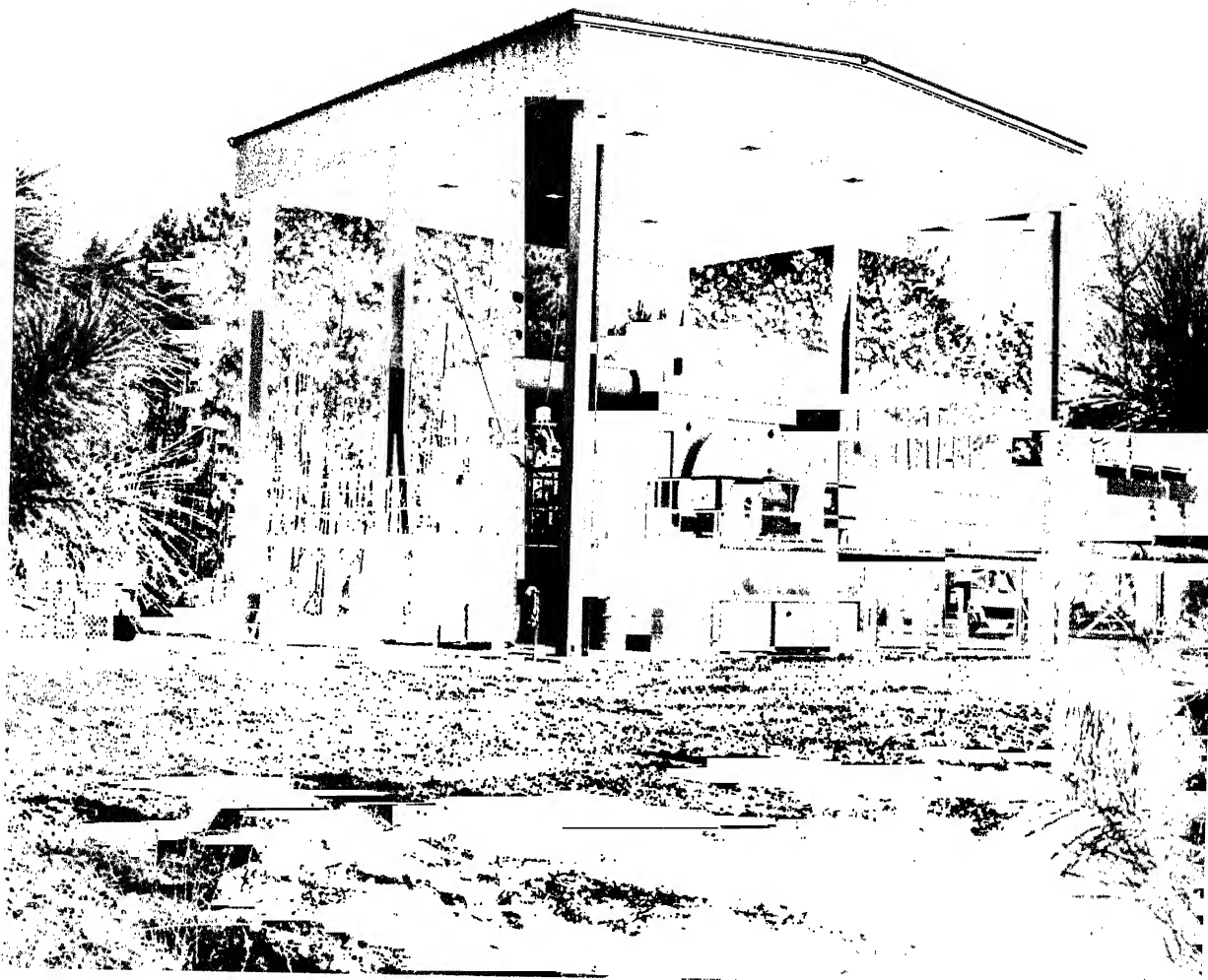
**RECENT WORK AND ACCOMPLISHMENTS** – Phases of the project are: dry combustion, simultaneous air/water injection, heat scavenging by water injection, and evaluation. The project area consists of five inverted 9-spot patterns on a 19-acre site. Combustion has been underway for more than a year, and oil production response has been sixfold to a present rate of over 400 bbl/d of oil. Simultaneous air-water injection was started in April 1977. The current injection rate is 965 bbl/d of water. The five patterns received air for the burn at a current rate of about 7 MMscf/d. Operations have included squeeze cementing of several producing wells to protect the downhole pump and tubing from high temperatures, conversion of the gas-driven air compressor to electric power, and treatments for scale buildup. Detailed chemical analysis of produced oil, water, and combustion gases have been performed. The analyses showed that the produced oil is upgraded slightly and that gas emissions are within acceptable limits.



*Subsurface Pressure Tests Aid in Designing and Operating In-Situ Combustion Project*

A set of pressure tests including falloff and pulse tests were performed to determine preferential directions of fluid flow. These tests aided in the location of a temperature observation well in the pattern. Monthly temperature surveys have detected the movement of the burn front a substantial distance into the formation in three patterns. A series of combustion tube tests was performed to determine optimum water-air injection ratio, which was estimated to be about 680 bbl/MMscf, but the burn was quenched when the ratio was 680 bbl/MMscf. Several dry burns

were conducted to determine ignition temperatures and to investigate low-temperature oxidation characteristics.



*Air Compressor for In-Situ Combustion To Recover Heavy Oil*

**PLANS FOR THE COMING YEAR** — Increases in water injection rate toward the optimum level are planned. To further investigate oxidation parameters and to refine heat-loss simulation, additional tube tests will be made. Reports covering the pressure-transient analyses, fluid properties, and combustion tube tests will be prepared. Methods of breaking emulsions will be tested, and technical and economic evaluations of the project will be made.

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## IMPROVED OIL RECOVERY BY STEAMFLOODING

GETTY OIL COMPANY  
DOE - \$2,000,000; Getty Oil - \$6,700,000  
6/25/76 - 5/24/81

**OBJECTIVES** – The purpose of this project is to demonstrate and evaluate, by a pilot operation, the feasibility of producing a highly viscous crude oil from an unconsolidated sand by steamflood in conjunction with short-term steam stimulations of producing wells. Many facets of steamflood technology will be demonstrated through the project; for example, completion techniques including in-place tensile prestressing of the casing to reduce thermal failure will be used. Also, reservoir performance and economics of the project will be compared with results obtained using a comprehensive mathematical model for thermal recovery. In addition, studies of the economics of scrubbing lease crude for boiler fuel vs use of sweet crude will be made. The site of the steamflood pilot demonstration is the SIB Sand of the Cat Canyon Oil Field in Southern California. This project will attempt to increase the recovery in the pilot area to 50 percent of the initial oil in place or 850,000 bbl. If the project is successful, an additional 100 to 120 MMbbl of oil will be recoverable from the Cat Canyon Field. Successful demonstration of steamflood recovery of such high-viscosity oil will encourage exploitation of over a billion barrels of heavy oil in other reservoirs.

**RECENT WORK AND ACCOMPLISHMENTS** – Phases of the work are preinjection, steam injection to breakthrough, steam displacement after breakthrough, and evaluation. The steamflood is being conducted at an average depth of 2300 feet. The flood pattern is four inverted 5-spot patterns in a 20-acre area. Steam stimulations of the production wells were conducted resulting in an average oil production of 200 bbl/d of oil. (Primary production in this zone is negligible.) In April 1977, steam displacement commenced and the injection rate averaged 2300 bbl/d of steam for the first 6 months. Generator downtime to remove and replace packers resulted in the equivalent of 3 months of injection during this period. Discharge temperatures and pressures varied up to 660°F and 2230 psi, which were found to be above the lower limits for packer failure. Accordingly, injection is now proceeding without packers as the resulting wellbore heat losses are deemed to be acceptable. Steam injection profiles indicated that the steam is entering the perforated interval with reasonable uniformity.

**PLANS FOR THE COMING YEAR** – The steam drive will be aided by periodic stimulations of the producers until steam breakthrough occurs. Project performance will be compared with proprietary three-phase, three-dimensional mathematical simulation of the steam-drive process. Improvements in the flue gas scrubber to be used with the steam generator and subsequent acceptance testing will be accomplished. A thorough economic evaluation of using and scrubbing high-sulfur lease crude oil vs sweet crude from another area as boiler fuel will be made.

## THERMAL OIL RECOVERY

HANOVER PETROLEUM CORPORATION  
DOE - \$951,725; Hanover Petroleum - \$2,096,334  
1/24/75 - 1/31/77

**OBJECTIVES** – This project was designed to demonstrate the efficiency and economics of recovering oil from a low-production reservoir by the combination thermal-drive process (forward in-situ combustion combined with water injection) in conjunction with high-temperature stimulation of

the production wells (in-situ combustion for a short time followed by production from the ignited well). The demonstration site was Little Tom Field, Texas. One of the most important phases of this work was the transfer of technology obtained from R&D to the private sector through DOE. Such technology obtained through performance should aid substantially in the selection of reservoirs suitable for EOR methods.

**RECENT WORK AND ACCOMPLISHMENTS** – The thermal-stimulation treatments in two wells during Phase I did not ignite the San Miguel Formation. Gas-analysis data indicate that low-temperature oxidation occurred, but no high-temperature burning zone was ever established. These treatments did not improve oil-producing rates, and casing was damaged in both wells.

The project was terminated as unsuccessful by mutual agreement on August 31, 1976. The final report was published in January, 1977.

**PLANS FOR THE COMING YEAR** – All technical aspects of this contract have been completed.

### **OIL RECOVERY BY COMBINATION THERMAL DRIVE**

HUSKY OIL COMPANY  
DOE - \$2,520,000; Husky Oil - \$4,791,000  
1/24/75 - 1/23/80

**OBJECTIVES** – Husky Oil Company is demonstrating the technical efficiency and economics of producing petroleum from a formerly low-productive reservoir using combination thermal drive (in-situ combustion utilizing controlled water injection) in conjunction with short-term thermal stimulation of production wells. The site is the Paris Valley Field near San Ardo in Monterey County, California. The reservoir, at a depth of about 800 feet, contains an extensive deposit of thick crude oil that falls between a true tar and a heavy oil. This crude oil (11 degrees API) is extremely viscous and cannot be produced economically without thermal stimulation. If the project is successful, other fields containing viscous oil may be exploited using combined thermal techniques.

**RECENT WORK AND ACCOMPLISHMENTS** – The work is being conducted in four phases: evaluation and preparation, dry combustion, combustion with controlled water injection and heat scavenging, and project evaluation. Approximately 3 months of combustion occurred before the compressor shut down because of damage in the cylinders. Although the burn time was too short to induce a meaningful production response, continued steam stimulation was effective. Oil production increased from a level of about 30 bbl/d of oil to a range of between 100 and 200 bbl/d. The variance resulted from the better response of the up-structure wells, following stimulation as compared to the down-structure wells. Producing water-oil ratio averaged about 10. Steam stimulations generally consisted of about 10,000 bbl of water as 72 percent steam. In one stimulation, air was added with the steam to add energy and reduce oil viscosity and thus increase the response. Radioactive steam profiles run in several wells showed that steam preferentially entered the lower portion of the Ansberry Sand's Lower Lobe.

**PLANS FOR THE COMING YEAR** – As soon as compressor repairs and testing are completed, combustion will be resumed. After a predetermined volume of formation has been covered, intermittent water injection will be started to redistribute fluids and heat for more advantageous oil

recovery. A recent contract expansion provides for drilling two temperature observation wells and periodic measurement of temperature profiles, pressure transient tests, and computer simulation of the steam stimulation process. In addition, further tests of the air-steam stimulation process will be made. These efforts, as well as completion and use of an automatic data collection system, will be pursued.

## HEAVY OIL/TAR SANDS, SOUTHEASTERN KANSAS

KANSAS GEOLOGICAL SURVEY (UNIVERSITY OF KANSAS)  
DOE - \$97,386; Kansas Geological Survey - \$33,662  
6/1/76 - 8/31/77

**OBJECTIVES** — This project sought to improve estimates of the location, size, and quality of heavy-oil deposits in southeastern Kansas as part of a coordinated study involving areas of Missouri, Kansas, and Oklahoma. Deposits of heavy oil in western Missouri and eastern Kansas occur at depths ranging from surface outcrops to more than 500 feet. Still farther south, in Kansas and in northeastern Oklahoma, other deposits occur at similar or slightly greater depths. The quality of these heavy oils is fairly good: their hydrocarbon content is moderately high and their sulfur content is low.

**RECENT WORK AND ACCOMPLISHMENTS** — To sample heavy-oil bearing sandstones in Bourbon, Crawford, and Cherokee Counties, Kansas, 33 test holes were drilled. Analyses of results of the drilling and geochemical analyses of the oils recovered have been completed. The main concern was to establish the quality of potential reservoir sandstones and the degree of oil saturation of these sands in the three-county area. Holes were drilled with air-rotary drilling equipment. Cores were taken by conventional rotary coring methods at selected intervals during drilling to sample the oil sands encountered. The 33 drillholes were scattered over about 1 million acres, which is fairly loose control. Hole locations were selected, however, on the basis of the need to evaluate shows in existing wells, or to gain control where none existed. The drilling program succeeded in recovering a wide enough range of representative materials from the subsurface as a basis for a fair assessment of the heavy-oil resources in the three-county area.

Approximately 1100 feet of sand were penetrated by the 33 test holes: 354 feet were cored, with 219 feet warranting analysis as oil-bearing; 130 feet contained an oil saturation greater than 15 percent, and 9 feet of the oil sand penetrated contained oil saturation greater than 50 percent; about 56 feet contained oil in amounts greater than 400 bbl/acre foot. The thickest sandstone with this much oil saturation was only 9 feet thick. Exploration for subsurface heavy-oil-bearing sandstones in southeastern Kansas has been less encouraging than anticipated. Even though sandstone in one or another of four or five zones was encountered in almost all of the 33 wells drilled, only a small fraction of this sandstone merited coring and analysis. Porosity, permeability, and oil saturation were all lower than expected in these sandstones. Shaliness of the sandstones and their discontinuity were other unfavorable factors. Further development of this resource may be warranted in selected portions of the study area but, in general, prospects for large-scale development of heavy oils in southeastern Kansas (east of the belt of presently producing oil fields) must be considered minimal.

**PLANS FOR THE COMING YEAR** — A final report has been prepared and is being processed for early publication.

## HEAVY OIL/TAR SANDS, WESTERN MISSOURI

MISSOURI DEPARTMENT OF NATURAL RESOURCES

DOE - \$79,328

6/1/76 - 12/31/77

**OBJECTIVES** – This study is being conducted to improve estimates of the location, depth, size, and quality of heavy-oil/tar-sand deposits in western Missouri and to determine the geologic setting, the reservoir characteristics, and the physical and chemical properties of the oil. Deposits of heavy oil in western Missouri and eastern Kansas occur at depths ranging from surface outcrops to more than 500 feet. Still farther south, in Kansas and in northeastern Oklahoma, other deposits occur at similar or slightly greater depths. The quality of these heavy oils is fairly good: their hydrocarbon content is moderately high and their sulfur content is low.

**RECENT WORK AND ACCOMPLISHMENTS** – Drilling operations in Missouri have been conducted in a two-phase program that allowed evaluation of first-phase drilling results before selecting drilling sites for the second stage of the drilling program. During the first phase (December 1976–April 1977) 21 tests were drilled and 3641 feet of section were cored. Total footage drilled was 3779 feet. Drilling sites were selected to obtain data where none existed and to validate existing shallow drilling in areas where only the upper portion of oil-saturated sandstones had been encountered.

Sandstone thicknesses of nearly 100 feet in one (more or less) continuous section were encountered. Oil shows ranged from a few feet of spotty saturation to more than 40 feet of continuous, but varying degrees of, saturation. Sandstone made up 1540 feet of the 3779 feet drilled, of which approximately 22 percent or 333 feet contained hydrocarbons. Three of the wells cored were chosen for selective sampling for core analysis. Nineteen samples were selected by visual inspection in an attempt to obtain data that would cover a wide range of oil saturations. Results of analyses showed the calculated total oil content to range from a low of 194 bbl/acre foot to a high of 1392 bbl/acre foot.

Drilling sites for the second phase (started June 1977) were selected on the basis of preliminary maps that showed: gross sand thicknesses, gross oil saturation, structure at the top of the Krebs or Bluejacket formation, and a configuration of the top of the Mississippian. These maps revealed definite trends in sand thicknesses as well as outlining areas of oil accumulation.

**PLANS FOR THE COMING YEAR** – The final stage of drilling, to be completed by November 1977, should further define these trends and provide data for resolving stratigraphic and correlation problems that will serve as a basis for valid conclusions on oil accumulations and projected resource base estimates. Cross-sections will be prepared showing correlation of strata as determined from core sample studies and electric logs of wells. Reservoir characteristics such as porosity, permeability, and oil saturation will be determined. Maps depicting numbers and thicknesses of sandstone bodies and the distribution of the oil saturation will be prepared. A final report will be prepared.

**HEAVY OIL/TAR SANDS, NORTHEASTERN OKLAHOMA**  
**OKLAHOMA GEOLOGICAL SURVEY (UNIVERSITY OF OKLAHOMA)**  
**DOE - \$121,152**  
**6/1/76 - 7/31/78**

**OBJECTIVES** – The objective of this project is to determine the location and areal extent, the volume and hydrocarbon content, and the economic value of the reported heavy-oil and tar sands in northeastern Oklahoma. Deposits of heavy oil in western Missouri and eastern Kansas occur at depths ranging from surface outcrops to more than 500 feet. Still farther south, in Kansas and in northeastern Oklahoma, other deposits occur at similar or slightly greater depths. The quality of these heavy oils is fairly good: their hydrocarbon content is moderately high, and their sulfur content is low.

**RECENT WORK AND ACCOMPLISHMENTS** – All records of occurrences of surface and sub-surface oil sands within the area of interest are being studied to make detailed correlations and evaluations by constructing structural maps, cross sections, and maps showing distribution and quality of the sands. The low-power microscopic examination of cores and samples taken from the 16 test holes drilled during the summer is completed. Work continues on thin-section preparation and petrographic examination of the sandstones penetrated, with the expectation of closer correlation of equivalent sands within the drilled area. The preparation of structure and isopach maps of the entire area is progressing. The initial phase of preparing approximately 61 cross sections has been completed and they are in various stages of the first draft. All require the addition of nomenclature and correlation of beds above and below the reference datum. Also in progress is a study of the numerous small oil and gas fields in Craig County where information is very sparse.

**PLANS FOR THE COMING YEAR** – Existing well data will be compiled, including all mechanical well logs, scout ticket information, and available well cuttings and cores. A detailed surface geological examination will be made, and the strata will be correlated with corresponding formation in southeastern Kansas. A three-dimensional framework of the rock strata of the lower Pennsylvanian sediments that may contain the heavy oils will be established by construction of structure maps, cross sections, and maps showing sand distribution and thicknesses and suggested or known oil saturations.

**STEAM-SOLVENT STIMULATION**  
**UNIVERSITY OF SOUTHERN CALIFORNIA**  
**DOE - \$10,000**  
**9/76 - 8/77**

**OBJECTIVES** – This study evaluated the potential use of nonvolatile solvents in enhancing the response of viscous crude-oil reservoirs to steam stimulation. Successful methods of reducing the viscosity in heavy oil reservoirs would increase the potential recovery. A small volume of solvent can significantly reduce viscosity of crude oil.

**RECENT WORK AND ACCOMPLISHMENTS** – To carry out the analysis, it was necessary to determine the viscosity of crude oil-solvent mixtures as a function of composition of the mixture



and the temperature. Such determinations were carried out in a falling-ball viscosimeter at temperatures up to 300°F. The reservoir model used was relatively simple, with the heated oil around the borehole flowing to the well under steady-state conditions. The performance of the reservoir following the injection of steam and following the injection of steam and solvent was compared. The solvent was assumed to mix uniformly with the viscous crude, and the amount of steam injected was reduced so as to achieve the same performance in both cases. The effectiveness of the solvent was measured by the reduction in the amount of steam required to secure the same enhanced production. The savings in the steam requirements was compared to the cost of the solvent. (It was assumed that the solvent would be purchased at market prices and sold at crude prices as part of the crude oil stream).

The results of the study indicate that there is no economic merit in the use of solvents along with steam in stimulating heavy oil reservoirs. The assumptions that were made in reaching the calculated results are considered to be overly optimistic in favoring the role of the solvent; therefore, the results indicating that the solvent does not provide any economic advantage are considered to be quite firm. This conclusion should not be extended to the potential use of solvents in oil well stimulation where the solvent is the primary agent for stimulation, nor to the potential use of solvents for enhancing certain aspects of steam drive operation. The role of volatile solvents and soluble gases, which in addition to affecting the viscosity of the reservoir fluids will provide reservoir energy, were not considered in this study.

**PLANS FOR THE COMING YEAR** – All work on this contract has been concluded. A report covering the work is in preparation.

## SECONDARY RECOVERY BY THERMAL METHODS

STANFORD UNIVERSITY PETROLEUM RESEARCH INSTITUTE

DOE - \$350,000

9/76 - Continuing

**OBJECTIVES** – The effects of elevated temperatures on rock properties are not readily understood. A fuller understanding would probably result in better design and evaluation of thermal-recovery projects. In heterogeneous reservoir formations, the steam-drive process is affected by gravity override and channeling through the highly permeable zones and consequent bypassing of the less permeable zones. The addition of an ancillary material along with steam may reduce bypassing and would thus increase vertical conformance and improve recovery. This study is investigating all the various conditions and procedures that may affect the performance of an in-situ combustion project, performing laboratory studies of fluid and rock properties at elevated temperatures and pressures, and investigating the use of ancillary materials to improve the steam-drive process.

**RECENT WORK AND ACCOMPLISHMENTS** – Equipment has been designed and constructed to measure oil-water relative and absolute permeability at elevated temperatures and pressures using actual reservoir rock and crude oil samples from California. A set of empirical correlations of high accuracy has been developed from the literature to relate some physical properties of sodium chloride brines as functions of temperature and salinity. Over 40 case histories of in-situ combustion field tests have been analyzed and are being correlated. A mathematical model of the gravity override phenomenon in in-situ combustion is being developed to facilitate the case-history analysis

effort. A combustion tube apparatus has been constructed to study the fuel and air requirements for field tests; the experimental results will be used to improve the in-situ combustion field-test correlations.

Over 70 technical papers and 100 patents have been reviewed to assess the feasibility of using foam as a permeability blocking agent in steam injection. Two different experimental setups have been designed and constructed to screen foaming agents, and a large number have been screened. In an attempt to describe the flow of foam through porous media, a new partial differential equation has been developed. An air permeameter has been designed and constructed to measure directional permeability in reservoir rock samples cut as spherical cores. A number of cores are being studied using this apparatus to assess the feasibility of this approach in permeability blocking experiments.

At the request of DOE, a technique has been developed and used to analyze a number of heavy California crude oils containing varying amounts of a specific solvent.

**PLANS FOR THE COMING YEAR** — The equipment for measuring oil-water capillary pressure and electrical resistivities at elevated temperatures will be designed. As many California heavy crude oils and reservoir rock samples as possible will be used in the relative permeability experiments. The results will be analyzed, disseminated, and some general correlations attempted. Experimental in-situ combustion data will be obtained on a number of California heavy oils. Mathematical modeling effort will be completed. The results of the experiment and mathematical modeling effort will be used to extend the correlations developed from field case histories of in-situ combustion. More field data will be acquired and analyzed.

The foam-screening experiments will be completed. A number of selected foaming agents will be used in laboratory displacement studies simulating steam injection. Work will be started on design and construction of a vertical sandpack model to test gravity override effects. This model will be used to study the effectiveness of various foaming agents in permeability blocking under simulated steam injection conditions. Mathematical study of foam flow through rocks will continue. The air permeameter experiment will be completed. A feasibility study for a possible field pilot test will be started.

## KINETICS OF UNDERGROUND COMBUSTION

UNIVERSITY OF TULSA  
DOE - \$25,000  
3/1/77 - 2/28/78

**OBJECTIVES** — Fireflooding offers a viable enhanced recovery technique for many U.S. oil reservoirs. Accurate mathematical modeling can help delineate the best prospects for this recovery method and aid in proper development of the reservoir during its application.

The kinetic reactions involved in forward combustion are to be studied through special sampling techniques during physical model experiments. A two-step mathematical model, involving coking of the crude oil and combustion of the coke residue, will use the data collected from the physical model to see if it satisfactorily represents the overall kinetics of forward combustion. Success will permit evaluation of the reaction rate constants modeling fuel laydown and burnoff,

and, hence, permit development of a physically realistic mathematical model for the entire combustion process.

**RECENT WORK AND ACCOMPLISHMENTS** – The lower flange of the existing high-pressure combustion tube was modified to accommodate a sampling probe. This probe, a 12-foot length of 3.32-inch O.D. stainless-steel tubing, can be inserted axially within the sand pack and withdrawn as the combustion front progresses down the tube. Tests proved that both liquid and gaseous samples of the fluid within the combustion tube could be obtained during a combustion experiment. A chromatography with mini-computer for simultaneous gas-liquid analysis was obtained, but several long delays were caused by malfunctioning. Eventually the mini-computer had to be replaced by the manufacturer. Calibration of the chromatograph is nearing completion. Programming of the mathematical model is about 50 percent complete.

**PLANS FOR THE COMING YEAR** – Experimental combustion tube runs will begin as soon as the chromatograph calibration is completed. At least six experimental runs will be made while gathering samples from the probe. These experiments will be made at three different pressures (100, 250, and 500 psig) and at a high and low flux rate. Data obtained from these experiments will then be compared to the results of the completed mathematical model.

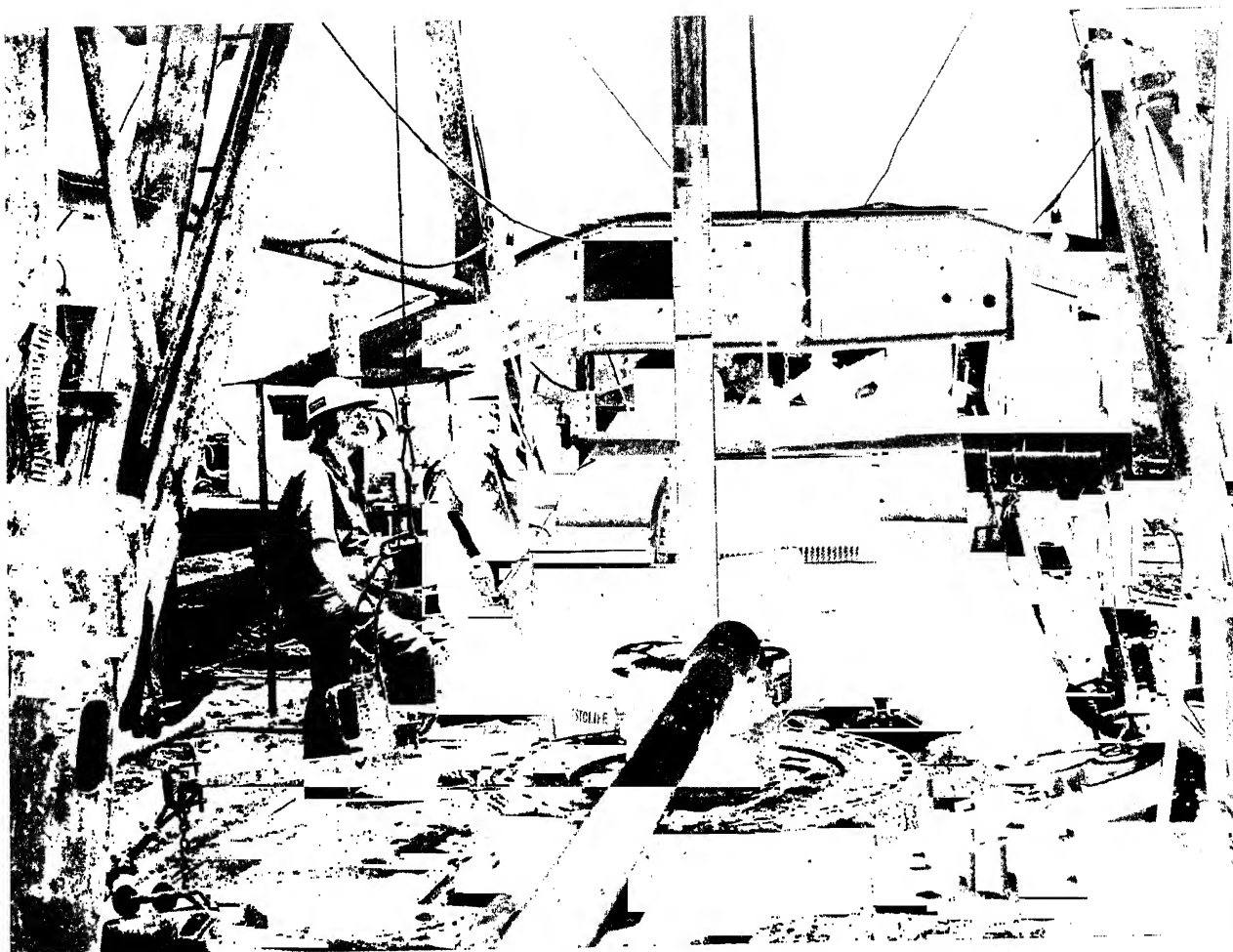
## **PROJECT HALO**

**BARTLESVILLE ENERGY RESEARCH CENTER**  
DOE - \$200,000  
1976 - Continuing

**OBJECTIVES** – The primary tool for discovery of small, shallow oilfields is still the drill. Soil-gas and related analyses are the only known methods that indicate the presence of subsurface oil or gas reservoirs. By combining these methods into a coordinated system with the more rapid magnetic and gravity surveys, which are capable of finding stratigraphic or structural traps that may contain hydrocarbons, a significant amount of unnecessary drilling may be eliminated.

**RECENT WORK AND ACCOMPLISHMENTS** – Field studies using radiometric and soil gas survey techniques were completed with a total of 1309 individual sites sampled. Four test wells were drilled in Kansas and two in Oklahoma to test the exploration procedure and obtain adsorbed hydrocarbon gas data at 10-foot increments from surface to an average depth of 2200 feet.

Laboratory efforts were directed to several areas in an attempt to relate soil parameters to surface-measured anomalies. A study determining the relative adsorptivity of generalized soil components when exposed to low-concentration paraffin gases was performed. Surface areas of soils used in soil gas determination were measured. Paraffin gases, extracted from drill cuttings taken from various depths, are being measured to determine if a pattern of hydrocarbon migration can be established between surface gases and oil or gas deposits at depth. Also, a carbon-isotope ratio determination is being made to establish whether the origin of the soil gases measured is biogenic or petrogenic. Computer data processing capabilities were consolidated and upgraded. The addition of digitizing and graphics hardware and the development of associated software allow data storage and manipulation directly from charts and maps.



*BERC Geologist on Location in Greenwood County, Kansas, To Collect Drill Cuttings for Evaluating Petroleum Exploration Methods*

**PLANS FOR THE COMING YEAR** — All field and laboratory work has been completed with the exception of analysis of drill cuttings from the six test wells. What remains is the compilation, integration, and evaluation of large amounts of data from both field and laboratory to determine the effectiveness of the combined exploration procedures studied. At the conclusion of that evaluation, the project will be terminated.

### **RESIDUAL OIL DETERMINATION**

**BARTLESVILLE ENERGY RESEARCH CENTER**  
DOE - \$400,000  
1977 - Continuing

**OBJECTIVES** — The accurate determination of oil saturation and its distribution in oilfields is essential for every petroleum reservoir considered for continuation of secondary recovery or implementation of tertiary recovery. This study will attempt to make such determinations of the magnitude and location of petroleum left in reservoirs during, or after, conclusion of current production practices.

**RECENT WORK AND ACCOMPLISHMENTS** – A workshop on oil saturation determination was held at BERC in April to provide a means for input from the public sector in identifying opportunities where DOE can focus its resources in oil saturation measurement. The workshop was chaired and paneled by representatives from industry and universities. To delineate the problem areas of residual oil determination properly, a series of key questions was compiled. The questions were divided into two major sections: fundamental research necessary to provide a sound basis for technological development, and field applications and activities. Both sections focus on the issues that are specific to DOE's residual oil determination programs within the framework of DOE's EOR Management Plan. After the workshop, a Technical Implementation Plan was prepared for inclusion in DOE's EOR Management Plan.

A review of the literature on residual oil saturation was made before the workshop; following the meeting, the notes and references were incorporated into a formal review of oil saturation determination which has been submitted for publication. Also, a paper was prepared detailing work showing that the calculated pressures and distributions of effluent particles from cores closely approximated the experimentally observed data when the transport process was treated as a random statistical phenomena.

**PLANS FOR THE COMING YEAR** – One or more field projects will be initiated that will compare, in a common reservoir, the currently accepted techniques for determination of residual oil saturation in that reservoir. This effort will involve a number of contracts for services with companies having particular expertise in using a specific measurement technique. In-house laboratory support will be provided as needed, and an effort will be made to develop means of overcoming weaknesses found to be inherent in any of the methods compared.

## ENVIRONMENTAL ASPECTS OF ENHANCED OIL RECOVERY

### BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$200,000

10/1/75 - Continuing

**OBJECTIVES** – This project seeks to determine the magnitude and duration of environmental damage that might possibly result from the injection of enhanced oil recovery chemicals into oil-bearing geologic formations. The specific objectives are to: determine the rates of migration of EOR compounds in the oil reservoir, establish the products of the natural degradation of EOR compounds in the subsurface environment, and determine the adsorption and chromatographic properties of EOR compounds at subsurface conditions of temperature and pressure.

**RECENT WORK AND ACCOMPLISHMENTS** – Computer programs to process laboratory data on dispersion (for determination of the coefficient of linear dispersion of miscible fluids in porous media) and for chromatographic transport, assuming equilibrium Langmuir adsorption isotherms apply, have been prepared, and experiments have begun. A third program for analysis of non-equilibrium rate-controlled chromatography is about 75 percent complete.

A thorough survey of technical publications related to micellar-polymer flooding operations is being conducted to determine analytic instrumentation and parameters of laboratory study that can be translated to field conditions. Liquid chromatography offers the best all-around analytic capability; therefore, an instrument was installed this quarter. The technical problems of the design

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and construction of apparatus for the determination of the degradation kinetics of EOR compounds at internal pressures up to 6000 psi and 100°C are gradually being worked out.

The migration of compounds in oil reservoirs after completion of EOR production project will be addressed by development of specific computer programs to scale laboratory data to field conditions and use them to predict the long-term pattern of migration of compounds in oil reservoirs of various types. The work was initiated by development of a frontal tracking program that will be further transformed by addition of parameters for dispersion, degradation, and chromatography.

**PLANS FOR THE COMING YEAR** – Laboratory experiments on the degradation kinetics of EOR compounds will begin as soon as six high-pressure high-temperature reaction vessels are assembled. The vessels will be used to simulate subsurface conditions of about 2000 meters. Gaseous products of degradation will be analyzed by gas chromatography and infrared. The heavier molecular weight components will be analyzed by several analytical techniques including liquid chromatography and infrared and ultraviolet spectrophotometry. Computer programs relating to migration, degradation, adsorption, and chromatographic properties of EOR compounds will be prepared.

#### DRILLING TECHNOLOGY RESEARCH PROGRAM

SANDIA LABORATORIES

DOE - \$450,000

4/1/76 - Continuing

**OBJECTIVES** – This program seeks to advance the development of drilling technology in the areas of high-performance bits, high-temperature-pressure drilling fluid instrumentation, and environmentally resistant materials. This program should have a major impact in reducing drilling costs, especially in deep wells, offshore, and in other frontier areas. The specific objectives for the present activities are: to develop an improved bonding technique for attaching the General Electric manmade diamond, Stratapax®; to develop a high-temperature-pressure field-portable mud viscometer; to improve the service life of existing elastomers with coatings and additives; and to determine the effect of heat treatment on the fatigue life of drill stem steels in a sour gas environment.

**RECENT WORK AND ACCOMPLISHMENTS** – An improved technique to attach the Stratapax to a bit has been developed. It utilizes an ion-beam metallization technique for coating the surfaces to be bonded with nickel. The Stratapax and stud (or bit) are then joined together with a gas pressure diffusion bonding process. This technique, which produces shear strengths of about 80,000 psi at working temperatures found in oil and gas wells, was refined in FY 1977 to improve manufacturability and reliability. Single point tests indicate that the bond has excellent resistance to heat, abrasion, and fatigue.

Two field portable viscometers have been built, and initial laboratory tests have been run to test instrument performance. One of the viscometers can test muds at various conditions up to 500°F and 20,000 psi, while the second viscometer is limited to 1500 psi at 500°F. Shear rates can be varied continuously up to 1000 sec<sup>-1</sup>. Elastomers have been coated with thin films to enhance their service life. Elastomers coated with parylene-C have shown some improved properties in lab tests. An improved RF Glow Discharge Process (plasma deposition) apparatus has been built, and the first coatings applied with this device show excellent adhesion. Tests to determine the effect of

heat-treatment techniques on fatigue life of steels in sour gas failed to show significant differences among various methods. They did show that fatigue crack growth in sour gas was severe even for steels with hardness below RC 22. It has been thought that steels below RC 22 were not as subject to sour gas embrittlement as harder steels.

**PLANS FOR THE COMING YEAR** — The bonding technique for the Stratapax will be made available to industry. Several bits will be built to demonstrate the ruggedness of the bond in laboratory and field tests. Further refinements in manufacturing techniques will be made. Additional testing of coated elastomers will be conducted, and compounding of elastomers with protective filler materials will begin.

## IMPROVED DRILLING AND CORING METHODS

### BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$60,000  
1975 - Continuing

**OBJECTIVES** — Improvements in well-drilling operations are sought that will decrease cost and time and will improve the quality of data obtained during drilling and coring. Methods to reduce drilling costs by increasing penetration rates will permit more drilled footage per year per dollar invested; thereby, oil and gas reserves will be increased.

**RECENT WORK AND ACCOMPLISHMENTS** — It was concluded from previous experimental drilling tests that a penetration rate loss of about 12 percent occurs when drilling hard formations at the surface and that a downhole liquid separator should be designed, built, and tested. A project to determine the effect of variable bit skew on penetration rate was initiated. The initial testing was completed, and the information is being analyzed.

**PLANS FOR THE COMING YEAR** — Future work will consist of monitoring the following contracts: Teleco, Inc. — Mud Pulse Telemetry; General Electric Co. — Electrodril with Telemetry; Sandia Laboratories — Improved Pressure Coring System; and Terra Tek, Inc. — Variable Bit Skew (Offset).

## PRESSURE CORING SYSTEM

### SANDIA LABORATORIES

DOE - \$225,000  
10/1/77 - 9/30/78

**OBJECTIVES** — A core barrel system is being designed that cuts a core with a minimum of drilling fluid invasion, and maintains the core at bottomhole pressure until examination. Such a design will result in much improved accuracy for the determination of in-situ reservoir fluids. As a consequence, fossil energy recovery will increase because of more efficient reservoir development and superior secondary and tertiary program planning.

**RECENT WORK AND ACCOMPLISHMENTS** — Investigations into the rock invasion properties of several silicon gels is underway. High-viscosity polymers have shown promise in laboratory tests on sandstone and limestone at pressure differentials up to 500 psi. These gels would be contained in a separate reservoir in the core barrel and would only be used to cut the core. Design concepts of



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various core bits have been initiated, and present work is favoring diamond-tungsten carbide disk Stratapax® cutters mounted on a bit configuration that is designed to reduce invasion by the drilling fluid. The mechanical core receiver design (by Maurer Engineering) has passed the review-study milestone, and work has begun on drafting layout studies.

**PLANS FOR THE COMING YEAR** – Laboratory tests of the core bit and the minimum invasion coring fluid will be conducted in rock samples utilizing a prototype in-house-fabricated Stratapax® core bit. Computer studies to optimize cutter location and bit hydraulics will be used in conjunction with these experimental data to finalize bit design and fluid selection. Detailed drawings of the pressure coring system will be completed.

## **ELECTRODRIL**

**GENERAL ELECTRIC COMPANY**  
DOE - \$1,024,000; GE and Industry - \$1,375,000  
5/1/76 - 9/30/77

**OBJECTIVES** – The goal here is to provide the petroleum industry with a modular electric drilling system in which a downhole electric motor is used to rotate the drill bit, and to obtain a hard-wire telemetry system for both directional drilling and conventional drilling. Phase I tested the directional drilling system, and Phase II is to test the deep drilling system. A reliable electrodril system will increase penetration rates, reduce well costs, and improve drilling safety.

**RECENT WORK AND ACCOMPLISHMENTS** – Phase I work was completed, and a final report is in preparation. Phase I results pointed out the need for some redesign work, and an Interim Phase was approved and performed between January and May 1977. This Interim Phase was set up to provide the transition between Phase I, Testing, and Phase II, Major Drilling System Test. Specifically, it included more detailed analyses, some redesign, and additional field testing required to rectify three problems that surfaced during Phase I: motor lower seal failures, excessive wear of bit shaft seal, and cable/connector failures. The reasons for failure was determined and corrective action was taken. A new replaceable electrodril connector was designed, fabricated, and tested. Repeated downhole matings were made during test. The cable and the replaceable male connector performed flawlessly. The internal conductor rings in the replaceable female connector gradually became oversize as the test progressed. Subsequent female connector rings will be fabricated using a harder, springier material to assure successful downhole mating even after extensive use.

Following the Interim Phase, a field test was performed at a wellsite in Houston. All systems performed satisfactorily for a short time until failure of minor components aborted the test. Work started on Task A of Phase II. Task A covers the engineering and pre-procurement work necessary to perform the field tests using the 285-hp deep drilling system outlined in the remainder of Phase II.

**PLANS FOR THE COMING YEAR** – Phase II, Task A work will be completed. If funds are available for the remainder of Phase II, the improvements discovered in Task A will be implemented. A solid-state telemetry system and remote-controlled heat sub for directional control will be tested. Improvements in high-temperature electronics will be made. The 285-hp deep drilling system will be tested in three deep wells and, in one of these tests, an experimental Stratapax® diamond bit will be tested.



## IMPROVED CORING FEASIBILITY STUDY

MAURER ENGINEERING, INC.

DOE - \$49,200

12/1/75 - 11/30/76

**OBJECTIVES** – The purpose of this work was to determine the feasibility of coring, retrieving, transporting and testing a reservoir rock sample while maintaining reservoir conditions on the cored samples; to devise a method to prevent the flushing of the core and thereby obtain the true saturation values of reservoir rock; and to improve the reliability of the existing pressure-coring equipment. These objectives are very important because more accurate saturation values may provide the key to predicting whether a tight gas sand would produce commercial quantities after massive hydraulic fracturing. Accurate saturation and pressure data would be valuable for evaluating oil reservoirs as enhanced recovery prospects or to check their efficiency by infield drilling in the flooded area.

**RECENT WORK AND ACCOMPLISHMENTS** – This feasibility study was completed during FY 1977, and a final report is being published. Some of the findings are as follows. Current formation evaluation techniques do not provide information of the accuracy desirable for making sound, economic decisions. This study analyzes the problem and identifies feasible alternatives for solutions. It concludes that a feasible solution is possible to provide about 90 percent information accuracy under many operating conditions. This accuracy is within the economic range for most oil and gas operations. This system is called Phase I in the study and should be pursued. The study also concludes that there is potential feasibility for the development of systems to approach 100 percent information accuracy under many operating situations; however, this type system is not practical within the economics of the oil and gas industry and would require several years plus many millions of dollars to build. It should not be pursued.

**PLANS FOR THE COMING YEAR** – The decision has been made to build and test the Phase I system. Sandia Laboratories will design an improved pressure coring system that will reduce the flushing of the core. If the designs are feasible, an RFP will be issued for a cost-sharing venture with an industry service company to build and test the Phase I tool. The system will include a pilot bit, a full gauge bit, a pressure-sealing core barrel, and will use a non-invading non-freezing gel material to surround the core, prevent invasion of mud filtrate, and extrude through the pilot bit for cuttings' removal and cooling.

## MUD-PULSE TELEMETRY

TELECO, INC.

DOE - \$2,000,000; Teleco - \$2,000,000

4/1/76 - 7/31/78

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type units and to build eight new tools for the pilot service demonstration. A goal of 250 hours without repair has been set.

**RECENT WORK AND ACCOMPLISHMENTS** — Equipment design was improved, and the prototype models were refurbished. The accuracy of the tool for measuring and transmitting downhole readings of borehole inclination, azimuth, and tool-face orientation was determined to be adequate. Eight new tools were built. They will be used to perform the pilot service demonstration. All field work was performed from offshore drilling platforms. More than 760 hours of downhole operation was logged for the prototype units. The measurements were proved accurate. The longest test before failure was 138 hours and the shortest was 15 hours, with an average test length of 82 hours. All failures were in minor parts that required only slight modifications. Teleco retired the prototype tools, and the pilot service demonstration (Phase III) started in August 1977 using the eight new tools. A Gulf Coast facility in Lafayette, La., was leased, remodeled and stocked. It is ready to provide support for the pilot service demonstration.

**PLANS FOR THE COMING YEAR** — The remainder of the program calls for servicing up to three offshore platforms. As many tests as possible will be performed in an attempt to obtain the desired 250 hours of reliability. After completion of the contract on July 31, 1978, the mud-pulse telemetry system is expected to be ready for commercial production.

## OPTIMIZATION OF DEEP DRILLING PARAMETERS

TERRA TEK, INC.  
DOE - \$267,173  
3/1/76 - 2/28/77

**OBJECTIVES** — This work simulated a deep-drilling environment in a laboratory and drilled rock samples with full-size bits to determine the optimum values for various drilling parameters. The relative importance of factors that affect drilling penetration rates was determined by taking data points while changing only one parameter and holding all other parameters constant. Drilling with full-size bits at simulated deep depths will be a significant contribution to petroleum industry drilling technology because formation rocks at great depths are under high in-situ pressures and are often in the ductile state.

**RECENT WORK AND ACCOMPLISHMENTS** — This work was completed in FY 1977, and the final report is being published. In this effort, full-scale laboratory drilling experiments were performed under simulated downhole conditions to determine what effect changing various drilling parameters had on penetration rate. The two rock types used for the tests were Colton Sandstone and Bonne Terre Dolomite. Drilling was performed with standard 7-7/8-inch rotary insert bits and water base mud. Variations in drilling parameters were: weight on bit—5000 to 40,000 lb; rotary speed—40 to 100 rpm; mud flow—80 to 220 gpm; mud pressure—100 to 5000 psi; and confining pressure on rock—0 to 9000 psi. Penetration rates from about 3 ft/hr to about 90 ft/hr were obtained.

The results showed the penetration rate to be strongly dependent on bit weight, rotary speed, and borehole mud pressure. There was only a small dependence on mud-flow rate. The drilling rate decreased rapidly with increasing borehole mud pressure for borehole pressures up to about

2000 psi. Above this pressure, the borehole pressure and rotary speeds had a smaller effect on penetration rate. The penetration rate was then dependent mostly on the bit weight. Penetration rate per horsepower input was also shown to decrease at higher mud pressures and bit weights.

The ratio of horizontal confining stress to axial overburden stress was maintained at 0.7 for simulated overburden stresses between 0 and 12,800 psi. For this simulated downhole stress state, the undrilled rock sample was within the elastic response range, and the confining pressures were found to have only a small or negligible effect on the penetration rate. Visual examination of the bottomhole pattern of the rocks after simulated downhole drilling, however, revealed ductile chipping of the sandstone, but more brittle behavior in the dolomite.

**PLANS FOR THE COMING YEAR** – The contracted work has been completed, and the results will be distributed to industry. Additional work needs to be done, but FY 1978 funding is inadequate to continue this study; however, it may be resumed in FY 1979.

#### OFFSHORE INSTRUMENTATION PROGRAM

SANDIA LABORATORIES  
DOE - \$396,000; USGS - 40,000; NOAA - 30,000  
7/1/76 - Continuing

**OBJECTIVES** – The overall objective is to develop reliable long-life instrumentation systems that will assist Government and industry in characterizing the environmental and engineering conditions of the outer continental shelf (OCS). The specific objectives of current efforts are the design, fabrication, and testing of a Seafloor Earthquake Measurement System. A sound state of knowledge of the geologic and marine hazards posed by frontier areas of the OCS is required if the recovery of the petroleum resources residing in these regions is to proceed in a safe and cost-effective way.

**RECENT WORK AND ACCOMPLISHMENTS** – Components common to a variety of seafloor instrumentation systems have been designed and tested. A high-data-rate acoustic telemetry system has been developed and successfully demonstrated in field tests conducted in Lake Mead, Nevada; the Gulf of Mexico, and Icy Bay, Gulf of Alaska. Digital data were transmitted through up to 600 feet of water at rates as high as 2400 bits/sec with error rates of less than 1 in 1000. During these field tests, a microcomputer-controlled data acquisition processing and storage subsystem was also evaluated. In addition, command and control of the seafloor instrumentation package was demonstrated. A limited number of temperature, pressure, and accelerometer sensors were tested. During the field tests, a known digital bit pattern was acoustically transmitted from a package installed on the ocean bottom to a surface receiving station. Three acoustic transducer beam patterns were tested and the transmission data rate and power level were varied to determine optimum system characteristics. The beam pattern, data rate, and power level were command-selectable from the surface receiving station. It was concluded from these field tests that a 90 degree beamwidth acoustic transducer operating at 4 watts presented optimal operating conditions for future systems.

**PLANS FOR THE COMING YEAR** – A prototype Seafloor Earthquake Measurement System will be designed, fabricated, and installed on the seafloor in an earthquake-prone region of the OCS, probably on the Southern California Coast. Sediment motion will be measured with force-balanced accelerometers capable of measuring accelerations from  $10^{-4}$  to 1 g. Data will be stored in a

solid-state nonvolatile magnetic bubble memory and retrieved, upon command, via a high-data-rate acoustic telemetry system. A Geotechnical Instrumentation Package will be designed also. This package will measure the response of key ocean-bottom sediment parameters, such as pore water pressure, hydrostatic pressure, and sediment motion cyclic loading resulting from shore waves.

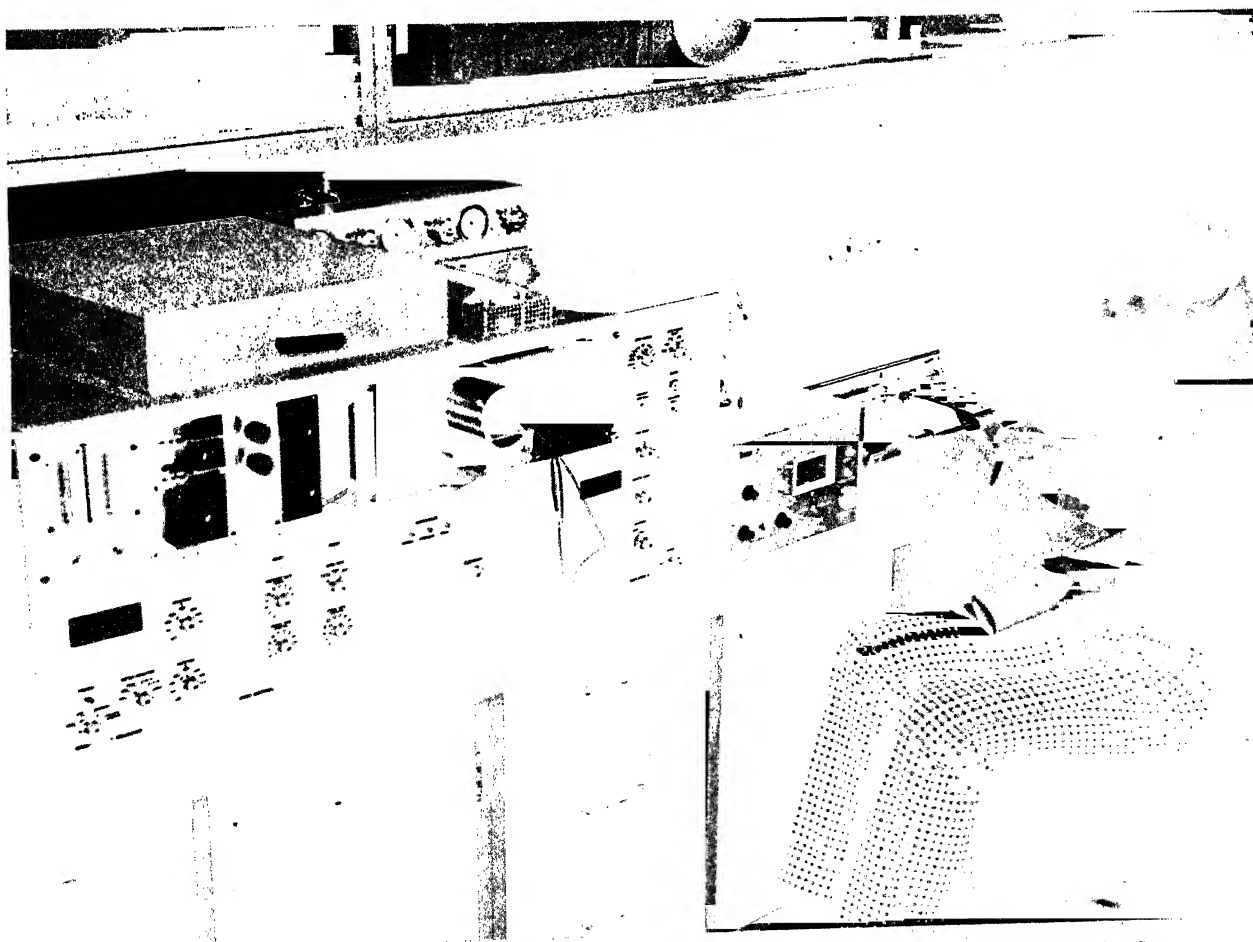
## CHARACTERIZATION STUDIES: HEAVY ENDS

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$250,000

7/1/74 - Continuing

**OBJECTIVES** – This project is designed to provide detailed characterization data for heavy oils, the heavy ends of petroleum, and similar materials as a basis for developing processes to upgrade these materials to more useful products. The data can also be used to provide correlations that can relate structural features of liquid components to geological origin and aid in production and processing of materials. Such data have not been readily available and will call for unique studies that will extend existing technology to these materials. Development and evaluation of advanced analytical techniques will also be necessary.



*BERC Chemist Uses Gas Chromatography with Nitrogen Detection Capability for Characterization Studies of Separated Chemical Classes of Petroleum Hydrocarbons*

**RECENT WORK AND ACCOMPLISHMENTS** – Separations were completed for five 535° to 675°C distillates thus providing the project with saturate, monoaromatic, diaromatic, and polyaromatic-polar concentrates from these high-boiling distillates; analytical techniques such as field desorption mass spectrometry (FDMS) and nuclear magnetic resonance (NMR) spectrometry are being examined as possible means of determining quantitative and qualitative distributions of components. A basic study in deriving gel permeation chromatography (GPC) parameters was developed and published. A characterization study of Bartlett, Kansas, heavy oil was published. A hydrotreated shale oil characterization study was completed. Experimental parameters continue to be explored in the preparation of new high-pressure liquid chromatographic (HPLC) columns that will be used for separating high-boiling distillates into saturate and aromatic concentrates in preparative scale quantities.

**PLANS FOR THE COMING YEAR** – Work will continue on high-boiling petroleum fractions that are available. Experiments with new HPLC columns will continue and probably will be completed during the coming year. FDMS and NMR will be further examined as to their applicability to the 535° to 675°C fractions available. Work delayed on acid- and base-removal will be resumed.

## CHARACTERIZATION OF HEAVY LIQUIDS FROM FOSSIL FUELS

LARAMIE ENERGY RESEARCH CENTER  
DOE - \$150,000  
7/1/74 - Continuing

**OBJECTIVES** – Methods will be developed to determine the composition and properties of the organic constituents of fossil fuel resources. These methods will then be applied to interpret and interrelate these data as they affect recovery, processing, and utilization. The goal of this project speaks to the problem of utilizing efficiently every fossil fuel energy resource. The design of efficient refining processes and the efficient utilization of fuel energy sources requires a knowledge of their composition. This project provides methods for obtaining this required compositional data.

**RECENT WORK AND ACCOMPLISHMENTS** – A separation method developed by the project provides valuable composition data by separating heavy distillates and residues into five chemically defined fractions: acids, bases, neutral nitrogen compounds, saturates, and aromatics. This method was applied to coal liquid, petroleum residue, and petroleum asphaltene samples. Characterization studies were made on fractions prepared from each of the samples. Optimum conditions were established for the separation of Synthoil coal liquid into fractions. Percentages of each fraction were: acids, 30 percent; bases, 20 percent; neutral nitrogen, 10 percent; saturates, 11 percent; and aromatics, 25 percent, with a total material recovery of 96 percent. Characterization of residues (material boiling above 675°C) from four crude oils was completed. A series of three papers is being prepared on the separation and characterization of petroleum residues. These papers describe (1) the separation methods used, (2) characterization of the compound types, and (3) a comparison of the composition of residues with that of high-boiling petroleum distillates. Asphaltenes from Wilmington, California, crude oil were separated and characterized to establish the relationship of acid and base compounds. A paper was presented on the composition and chemistry of petroleum asphaltenes at the American Chemical Society meeting in March 1977. A meeting on Fossil Fuel Chemistry and Energy (Confab 77) was arranged and hosted in July 1977. Eighty scientists from academic, industry, and government laboratories attended the program that consisted of 30 technical papers.

**PLANS FOR THE COMING YEAR** – A paper on the separation and characterization of Synthoil will be prepared and presented at the American Chemical Society meeting in March. Coal tars produced from the Hanna IV underground coal gasification experiment will be examined for compound composition. Three papers on the composition of petroleum residues will be completed and published as journal articles. A study will be initiated on the heavy distillates and residues of shale oils. Four shale oils, representing aboveground and in situ retorting processes, will be separated and characterized according to compound-type composition.

## **QUALITY OF CRUDE OILS AND PRODUCTS**

**BARTLESVILLE ENERGY RESEARCH CENTER**  
DOE - \$120,000; American Petroleum Institute - \$18,000  
10/71 - Continuing

**OBJECTIVES** – This project provides detailed characterization and physical property data relating to the quality of U.S. petroleum reserves, imported crude oils, and of petroleum fuels marketed in the United States. The continuing input of new crude-oil data, combined with a viable working file of about 9000 analyses, provides an extensive data base for use by Government, industry, and university in developing improved crude-oil recovery techniques and utilization for various petroleum products or petrochemical stocks. Periodic surveys of nationwide fuel quality for motor gasolines, aviation turbine fuels, diesel fuels, and heating oils provide a basis for determining important trends as they occur and for determining needs for future improvements in engine design and/or fuel quality.

**RECENT WORK AND ACCOMPLISHMENTS** – The analysis of over 75 oils has provided new insight for crude oils from important new U.S. and various foreign oil fields. These analyses also included data for a number of important coal liquids and for oils involved with special production research on tertiary recovery techniques. This project received large quantities of fuel quality data through a cooperative working relationship with the American Petroleum Institute and published as Petroleum Products Surveys covering summer motor gasolines, winter motor gasolines, aviation turbine fuels, diesel fuel oils, and heating oils. Over 1000 individuals in Government, industry, and universities received these five publications for their use in improving fuel quality and engine design. A comprehensive survey of 800 important U.S. producing oil fields and/or differing formations within a field is nearing completion.

**PLANS FOR THE COMING YEAR** – A substantial number (75 to 100) of crude oils and syncrudes will be analyzed to provide much needed data for improved utilization. The development of analytical techniques and procedures as well as improved laboratory equipment and facilities will provide even more needed data. Plans include the introduction of all crude-oil data into computer storage for easy accessing of specific data needs involving all areas of DOE research as well as those of industry and universities. Properties of motor gasolines, aviation turbine fuels, diesel fuel oils, and heating oils marketed in the United States will be surveyed, published, and distributed in this country and abroad to interested individuals in Government, industry and universities.

## OIL IDENTIFICATION

BARTLESVILLE ENERGY RESEARCH CENTER  
DOE - \$200,000  
1972 - Continuing

**OBJECTIVES** – This research determines, measures, and computerizes distinctive properties and related data on crude oils and crude-oil residues and establishes reliable definitive interrelationships of these diverse properties for improved oil “fingerprinting” and identification. Such distinctive properties and definitive interrelationships are widely applicable and are used in the areas of oil pollution and geochemical exploration.

**RECENT WORK AND ACCOMPLISHMENTS** – Infrared absorbance data were obtained for 52 selected artificially weathered (distilled through 275°C) crude-oil samples. Statistical analysis of the resultant data and comparison with data obtained on the unweathered crude oils were completed. Similar statistical analyses of weight percent distributions and even-odd predominance distributions for normal paraffins, obtained by gas-liquid chromatographic (GLC) analysis on a statistical population—52 topped crude-oil samples, were used to evaluate these distributions for oil identification.

Three techniques for analysis of nickel and vanadium in crude oils and crude-oil residues by both flame- and flameless-atomic absorption (AA) spectroscopy were evaluated for reproducibility and accuracy. Ninety crude oil and residuum samples were analyzed for Ni and V using a flameless AA technique. Flame photometric detector responses for a wide variety of sulfur-containing compound types were measured and used to obtain sulfur weight percent distributions for 28 selected crude oils and corresponding topped crude-oil samples by high-resolution GLC. Detailed statistical analysis of these distributions indicate that they are of significant value to oil identification when used in conjunction with n-paraffin distributions. A GLC procedure was developed and evaluated for the reliable determination of benzene and toluene in crude oils and related material.

A contractual study for carbon, nitrogen, and sulfur stable isotope data was initiated with Global Geochemistry, Inc., and UCLA. Low-temperature total luminescence contour spectra were evaluated for oil identification.

**PLANS FOR THE COMING YEAR** – Exploration of high-resolution GLC analysis of crude oils and crude-oil residues will be continued with emphasis on normal and isoparaffin distributions, sulfur-containing compounds and their distributions as analyzed by flame photometric detection, and nitrogen-containing compounds and their distributions as obtained by thermionic detection. Trace metals analysis will be expanded to include a wider range of metals. Utilization of mass spectrometry for oil identification will be evaluated using both conventional electron impact and field ionization. The IR spectrophotometer and GLC will be interfaced to a minicomputer and suitable software written to facilitate data acquisition and analysis. The stable isotope ratios of sulfur, nitrogen, and carbon will continue to be studied. The differentiation of highly similar crude oils will be attempted using a multianalytical approach.

## REFINING PROCESS TECHNOLOGY

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$161,000

6/1/75 - Continuing

**OBJECTIVES** – This project is determining the refining characteristics of synthetic crude oils in relations to source, composition, and type of finished products, and establishing overall refining processes needed to satisfy future patterns of usage of refined liquid fuels. Synthetic crude oils, especially those derived from coals, are expected to require greater severity of refining than typical petroleum, and the correlations sought will help determine the changes in refining processes necessary to achieve efficient and economical production of refined fuels.

**RECENT WORK AND ACCOMPLISHMENTS** – Design of a bench-scale hydrogenation refining unit for generation of comparative data on the initial stages of refining synthetic crude oils was completed and a contract let for assembly. Samples of liquids from several coals, ranging from bituminous-A to lignite, were prepared and upgraded by catalytic hydrogenation in a batch autoclave at mild reaction conditions. Coals of lower rank were more reactive and the liquids more easily upgraded, but the nitrogen contents of all were decreased to about 0.2 weight-percent to permit determinations of hydrocarbon type as a function of coal source in another project. State-of-the-art studies were made on the refining of shale oil and on the denitrogenation of coal liquids, with principal background coming from the refining of petroleum.

**PLANS FOR THE COMING YEAR** – Preparation of the series of liquids from a wide range of coals will be completed. The bench-scale refining unit will be installed, operation checked out at pressures up to 3000 psig hydrogen, and initial tests made on a synthetic crude. Studies of future refining technology will be continued with the use of available composition and processing data.

## WASTE OIL RECYCLING

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$350,000

1972 - Continuing

**OBJECTIVES** – This research seeks to develop and evaluate new or modified technology for reclaiming used lubricating oils, obtain compositional data on lubricating oil basestocks and/or feedstocks as a basis for upgrading reclaiming technology, demonstrate technical and economic feasibility of the process found most promising, extend successful technology to the reclamation of selected industrial oils, and develop useful roles for byproducts derived from these processes. Advanced technology for efficient, economical, and clean re-refining of used automotive lubricating oils is required to stimulate the re-refining industry, thereby conserving natural petroleum resources and reducing environmental pollution. Over three times the energy is required to produce a barrel of lubricating oil from crude oil than by re-refining a barrel of used lubricating oil.

**RECENT WORK AND ACCOMPLISHMENTS** – The BERC staff produced the first U.S. documentation of the substantial equivalency of re-refined and virgin-derived automotive lubricating oil as determined by engine sequence tests. Recognition for research excellence in the field of lubricating oil reclamation was accomplished, and close personal relations have been established with European



leaders in used lubricating oil re-refining. A small contractual study in Iowa supported in part from BERC research funds has established the successful performance of re-refined automotive lubricating oils in a fleet test of 50 vehicles, half of which are using re-refined and the others virgin-derived lubricating oil. The BERC solvent/distillation process for the reclamation of used automotive lubricating oils was granted U.S. patents and applications are pending in six foreign countries. The process is the only new technology that has documented, through publications, technical viability; economic studies indicate the operating costs to be more favorable than for the prevalent acid/clay process.

Present work includes the processing of 1000 gallons of used oil by the BERC-developed technology; a detailed study to investigate engineering parameters related to design considerations for a large-scale demonstration plant; and the first phase of a closed-loop composition study which is nearing completion.



*BERC Scientists Adjust Equipment Used for Research on Re-Refining Waste Lubricating Oils*

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**PLANS FOR THE COMING YEAR** – Complete processing of 1000 gallons of used automotive lubricating oil and successful engine performance tests of the product are anticipated. High on the list of priorities is funding approval for the design, construction, and operation of a 10-million gal/yr demonstration plant to prove economic and technical viability of the BERC solvent/distillation process. A cost-sharing partner from the private sector will be selected when funding is assured. In anticipation of this goal, engineering studies are being performed on a pilot plant scale to define an appropriate design for such a plant. Closed-loop composition studies will be continued and if funds and personnel permit, a study of byproduct utilization and disposition will be initiated.

### **IOWA RE-REFINED OIL FLEET TEST**

**IOWA STATE UNIVERSITY**  
DOE - \$50,000  
1976 - Continuing

**OBJECTIVES** – This fleet test was designed to obtain comparative performance data on re-refined and virgin 10W-30 motor oils and hydraulic oils in fleet use. One ultimate goal of the research will be the determination of economic feasibility of using re-refined lubricating oil in all Iowa state vehicles. The vehicles used in these tests were provided by the Iowa Department of Transportation. The re-refined oils were the commercial product from Motor Oils Refining Company and the experimental product from the Bartlesville Energy Research Center (BERC) derived from new technology developed at BERC.

**RECENT WORK AND ACCOMPLISHMENTS** – Forty-six vehicles including station wagons, pickups, and trucks with hydraulic systems were selected for operating in normal use with one vehicle on virgin-derived oil for each vehicle on re-refined oil. Oil change samples are analyzed by standard ASTM methods; 4000-, 8000-, and 10,000-mile oil-change intervals are being used. All vehicles were operational in this test program by September 1976.

All test oils were performing satisfactorily at the 4000-, 8000-, and 10,000-mile drain intervals, and no significant differences were noted among the three oils; therefore, 12 of the vehicles on 4000-mile drain intervals were changed to 8000-mile drain intervals to increase the stress on the oils. The oil is drained at appropriate intervals and standard ASTM analytical methods including flash, viscosity, insolubles, total acid and base number, fuel dilution, antifreeze, water, and metals analysis are performed. The vehicles are operated under a variety of conditions ranging from high-speed/high-load to stop-and-go with high idling time.

After 12 months of the 2-year program, preliminary results showed the re-refined oils were performing satisfactorily in extended oil-drain operation. The total base number of the virgin oil was substantially lower than that of the re-refined products, but no operational difficulties were observed as a result. The most serious observation from these tests is that operating performance of an oil may not be adequately insured by any bid specifications in use today, whether the oil is from virgin or re-refined stock.

**PLANS FOR THE COMING YEAR** – Contractual obligations to DOE will be completed. At the end of 2-year fleet testing period, 12 engines will be selected from those that complete the test, and deposit and wear ratings will be performed. Southwest Research Institute will assist in selecting the

12 engines and in performing the ratings. The cumulative oil analyses and the deposit and wear ratings are expected to provide an adequate comparison of the oils. A lack of significant differences among the three oils would be a useful result and would permit the Iowa Department of Transportation and other state agencies to allow re-refined oil producers to bid for the state's business. It may also encourage environmentally superior use of waste oil in Iowa. BERC will use the data provided from these tests to demonstrate and support the technical viability of the re-refining technology to produce high-quality motor oil from used crankcase drainings.

## STABILITY CHARACTERISTICS OF HYDROCARBON FUELS

BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$100,000

1976 - Continuing

**OBJECTIVES** — This research project is determining the storage stability characteristics of representative liquid hydrocarbon fuels, which involves detailed analyses of various gasolines, diesel and jet fuels including determinations of hydrocarbons and nonhydrocarbons that are found to be precursors of deterioration products. Ultimately, this information will guide refiners in selecting processes to minimize deterioration through removal or inhibition of reactive compounds. This work will be especially valuable to syncrude refining by predicting those compound classes that most need to be avoided. A near-term objective is to evaluate the storage and thermal stability of commercial fuels across the country.

**RECENT WORK AND ACCOMPLISHMENTS** — Routine physical analyses and accelerated aging tests have been contracted to U.S. Army Fuels and Lubricants Research Laboratory, Southwest Research Institute, San Antonio, Texas. All physical analyses have been completed for 6 fuels from petroleum and 14 synfuels from coal, oil shale, and tar sands, while accelerated aging tests are now underway. An atomic fluorescence system has been assembled for multielement trace metals analyses of the fuels being studied. This system has been calibrated with commercial standards and is now ready for use.

A meeting was held at BERC on March 9 to bring together some 25 members of industry, Government, the military, and education with a common interest in stability and fuels derived from alternate sources. The meeting served to define the state-of-the-art, encouraged the informal exchange of ideas, enhanced cooperation, and should help avoid duplication of effort.

**PLANS FOR THE COMING YEAR** — In addition to completing the original contract work on the 20 fuels described previously, a new contract is to be written with Southwest Research Institute involving a national fuels survey that will entail obtaining samples of leaded and unleaded gasoline and diesel and jet fuel from 12 sites across the country. These samples will be analyzed in detail for potential stability problems. Several analytical tools will be evaluated for their responsiveness to fuel deterioration products and precursors to deterioration. Correlations of stability to viscosity, GC spectra, and HPLC spectra are planned.

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## TAR SANDS CHEMISTRY

LARAMIE ENERGY RESEARCH CENTER

DOE - \$320,000

1972 - Continuing

**OBJECTIVES** – This project supports engineering research on recovery of energy from tar sand deposits by characterizing products (oils, gases, chars, water) from laboratory and field recovery experiments; evaluates products with regard to refinability and environmental impact; and evaluates the properties of tar sand bitumens that affect extraction and recovery.

**RECENT WORK AND ACCOMPLISHMENTS** – Analyses of oils and waters produced during the first in-situ combustion field experiment were essentially completed. Much of the product oil was very similar to original bitumen in both physical properties and chemical analysis. About 30 percent of the product oil was a lighter oil, most of which (more than 90 percent) distilled in the gasoline-naphtha-kerosine range. Waters contain mainly carboxylic acids as organic contaminants and ammonium sulfate as the major inorganic contaminant. Before initiation of the second in-situ reverse combustion experiment, bitumen from core samples was analyzed thoroughly. This bitumen had lower wax content and pour point than bitumen from the first site. Laboratory pyrolysis of core samples from the second site yielded oils of lower pour point than were observed upon pyrolysis of bitumen from the first site.

Pyrolysis studies of four Utah and Athabasca whole tar sands showed that temperature and feedstock affect product yield and composition. A survey is continuing to determine which tar sand bitumens contain high-molecular-weight hydrocarbons that contribute to high pour points of the bitumens or oils produced from them. Identification of these materials will aid in classifying bitumens that may cause handling problems during production. Bitumens are also being analyzed for vanadium and nickel contents because these materials cause problems during refinery operations. Oil, gas, and water samples from the second field test are being processed as they are collected. Although very little data are available, preliminary values indicate similarities with products from the first field experiment.

**PLANS FOR THE COMING YEAR** – Extensive analysis will be provided for products from the second field experiment. These data will be used to aid in obtaining material and heat balances for the run. Core samples from other domestic tar sands deposits will be evaluated as they are available in order to extend the data base for these domestic deposits. Kinetic and analytical data will be obtained on cores from sites proposed for the third field recovery experiment to aid in a rational selection of a site.

## UTILIZATION OF ALTERNATIVE FUELS—METHANOL

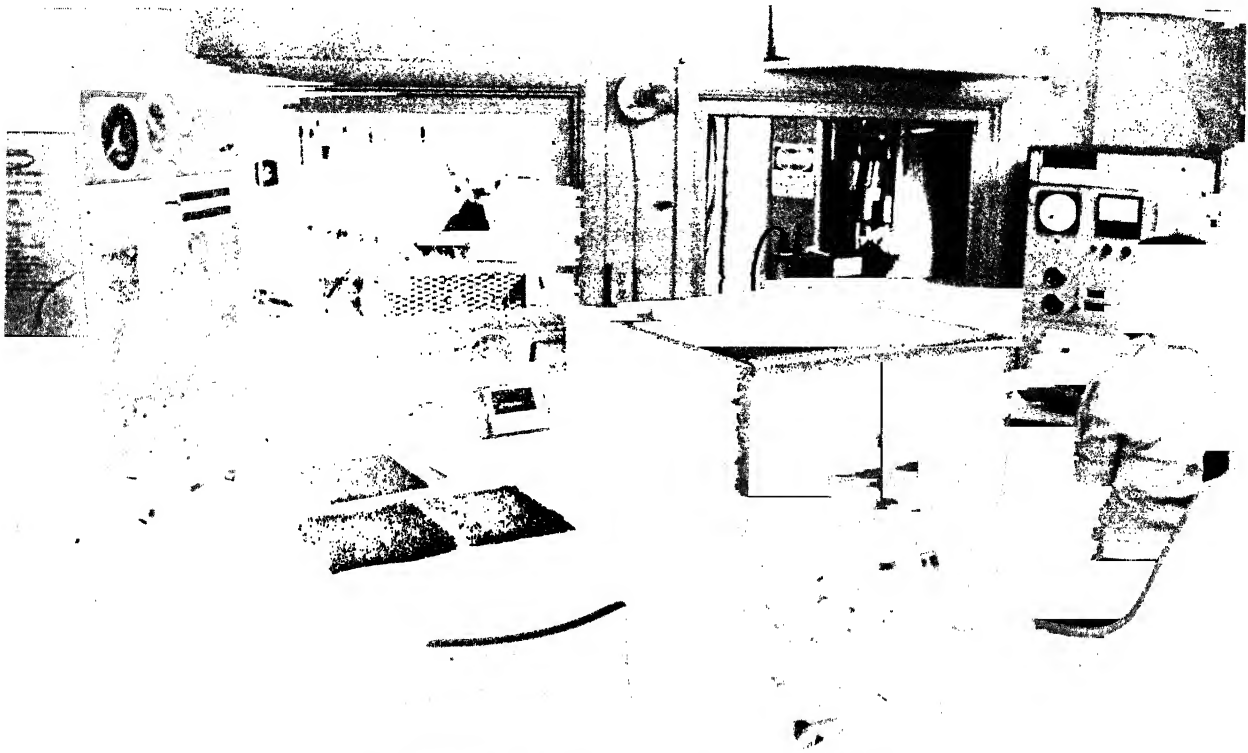
BARTLESVILLE ENERGY RESEARCH CENTER

DOE - \$350,000

6/1/75 - Continuing

**OBJECTIVES** – This project is obtaining experimental data on physical, chemical, and combustion properties of methanol for a comprehensive engineering assessment of problems and opportunities in its use as motor fuel. This work provides technical data to support analysis of energy-

development options and basic engineering data required for design, alignment, and parametric adjustment of fuel/engine systems as appropriate for favorable efficiency in using methanol and/or methanol-gasoline fuels.



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*BERC Technicians Measure Fuel Economy and Emissions of Engine Running on Methanol*

**RECENT WORK AND ACCOMPLISHMENTS** — Extensive experimental work has been done on the properties of methanol and methanol-gasoline blends including engine fuel economy and emissions characteristics. Current work, scheduled for completion in FY 1979, will complete acquisition of the principal engineering data needed in evaluating automotive-use options applicable to current technology engines and vehicles.

**PLANS FOR THE COMING YEAR** — Experimental and analytical work will be done (a) to measure fuel economy, emissions, and related engine-performance characteristics for combinations of methanol-gasoline fuel mixtures and engine parametric adjustments by use of modified current technology engines and prototype and development models of new lean-burn and stratified-charge engines; (b) from the data of (a), to develop the relationships of performance factors to design and adjustment variables; (c) to determine compatibility of automotive fuel components with methanol fuel blends via extended use in vehicle fleet tests; (d) to investigate additive materials that would increase the water acceptance of methanol-gasoline mixtures, and (e) to provide engineering-evaluation data for new fuel and fuel-air metering systems required for successful adaptation of automotive equipment to methanol fuels.

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## INCREASING USEFUL LIFE OF ASPHALT

LARAMIE ENERGY RESEARCH CENTER  
DOE - \$150,000; Federal Highway Administration - \$80,000  
1969 - Continuing

**OBJECTIVES** – This project is providing information on chemical composition, reactivity, and composition-property relationships of petroleum and synthetic asphalts from shale oil, coal syncrudes, and tar sands to conserve their energy value and improve their utilization; and determining the optimum chemical properties of softening agents for the reclamation and recycling of asphalt in wornout roads. Energy shortages dictate that materials with fuel potential such as asphalt be conserved. A 15 percent increase in road life could conserve the equivalent of 25 million barrels of oil annually. In addition, recycling 50 percent of the roads now needing replacement would save 45 million barrels of oil annually.

**RECENT WORK AND ACCOMPLISHMENTS** – Research is aimed toward the solution of two major problems in asphalt roads: environmentally induced failure of the adhesive bond between asphalt and aggregate, which results from the influence of water, and the oxidative hardening of the asphalt binders, which leads to premature failure of asphalt pavements and other asphalt products. The oxidative hardening studies include compatibility of asphalt components, which is related to durability. In asphalt-aggregate bonding studies, the relative tendency of the polar asphalt molecules forming the monomolecular layer on the aggregate surfaces to be displaced by water was determined. Although carboxylic acids are most strongly adsorbed on aggregates, they are most readily displaced. Nitrogen compounds, ketones, anhydrides, and carboxylic acids were shown to migrate to the asphaltene fraction on aging. Model studies showed that polyaromatic compounds with alpha-branched alkyl groups on adjacent bridgeheads readily form anhydrides, which is consistent with the proposed mechanism of anhydride formation in asphalt. The chemical properties of shale-oil asphalts are being characterized. A series of aged asphalt road materials is being recycled in laboratory studies using liquids from shale oils, coal syncrudes, or petroleums as rejuvenating agents. A screening test is being developed to select the best combination of asphalt and recycling liquid. Fundamental chemical and physical data gained in asphalt-aggregate interaction studies provided an explanation for a serious tender road-mix problem in Utah. A cooperative study with the University of Idaho showed correlations between their test for moisture damage and LERC data on basic chemistry of the asphalt-aggregate interaction. Studies of aggregates immersed in asphalts in an ultrasensitive microcalorimeter showed a good correlation between low heats of immersion (adsorption of polar groups on aggregates) and the tendency of an asphalt to produce a tender or flow-setting road mix.

**PLANS FOR THE COMING YEAR** – Work on evaluating asphalts from shale oil will continue. Engineering and chemical properties of road mixes prepared from a mildly hydrogenated shale asphalt will be evaluated. A program on the evaluation of heavy liquids from coal syncrude, shale oil, and petroleum as softening agents to reclaim wornout roads is planned. Engineering properties of selected materials from the recycle program will be conducted. Research will be performed on basic asphalt molecular interactions that lead to asphalt failure.

## FOSSIL FUEL CHARACTERIZATION BY MASS SPECTRAL TECHNIQUES

OKLAHOMA STATE UNIVERSITY

DOE - \$108,675

6/9/75 - Continuing

**OBJECTIVES** – Qualitative and quantitative information will be obtained concerning the composition of fossil fuels using the technique of mass spectrometry. Such compositional information is important in evaluating synthetic fuel conversion processes and process conditions, determining appropriate refining processes, and selecting best and most efficient end-use products. Specific mass spectral techniques to be employed include low- and high-resolution field ionization mass spectrometry and high-resolution electron impact mass spectrometry. Construction of a field desorption mass spectrometer source will be followed by application to the analysis of relatively nonvolatile, high-molecular-weight fossil fuel components.

**RECENT WORK AND ACCOMPLISHMENTS** – A study of the effect of ion source temperature on relative field ionization (FI) sensitivities of saturated and aromatic hydrocarbons was completed. Increasing the ion-source temperature from about 100° to 300°C reduces the dependence of saturate/aromatic relative FI sensitivities on mixture composition to within the limits of data precision. Relative sensitivities for FI of saturated hydrocarbon mixtures at 270° to 300°C ion-source temperatures were found to be independent of composition of the saturate mixtures. Development of a field desorption source and related accessories was completed, and some preliminary experiments on emitter conditioning were conducted. High-resolution 70-eV electron impact mass spectra were recorded for 56 fractions from separation of a tar sand sample on photographic plates, and data reduction and interpretation were started.

**PLANS FOR THE COMING YEAR** – Characterization of the tar sand sample will be completed. Applications of the field desorption mass spectral technique to high-boiling petroleum fractions will be initiated. Development and applications of FI mass spectrometry will continue. The project has been expanded to include the development of computer software for the reduction, correlation, and interpretation of experimental mass spectral data especially as related to characterization of fossil fuels.

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## *ENHANCED GAS RECOVERY*

This program of research, development, and demonstration (RD&D) for enhanced gas recovery (EGR) includes cost-shared contracts with industry and in-house research at DOE Energy Research Centers at Bartlesville, Oklahoma (BERC) and Morgantown, West Virginia (MERC). Projects also are conducted at Oak Ridge National Laboratory (ORNL), Los Alamos Scientific Laboratory (LASL), Sandia Laboratories (SL), Lawrence Livermore Laboratory (LLL), and Mound Laboratory (ML). Supporting research is performed at numerous universities. Rapid technology transfer is emphasized and is effected through periodic symposia, quarterly contract reports, in-house quarterly research reports, and technical presentations and publications by both DOE and contractor personnel.

The EGR program includes the production of natural gas from four unconventional sources: western tight (low permeability) sands, eastern tight shales, coal beds, and geopressed aquifers. Production-stimulation technology used includes advanced (massive) hydraulic fracturing, chemical explosive fracturing and combinations of the two, and wells deviated to intersect natural fractures. Currently, DOE has 15 cost-shared contracts with industry for gas-production-fracturing-stimulation field tests at a total cost of \$31.1 million, with industry funding 46 percent of the total.

Resource evaluations are made by the U.S. Geological Survey, universities, and state geological surveys. Supporting research is performed at the institutions listed above.

Goals are, by 1985, an addition of 10 trillion cubic feet to proved reserves and an incremental daily production increase of 3 billion cubic feet. Project management for the Eastern Gas Shales Project is delegated to the Morgantown Energy Research Center and that for the Western Gas Sands Project to the Bartlesville Energy Research Center.



*Massive Hydraulic Fracturing for Eastern Gas Shales Program*

## EASTERN GAS SHALES PROJECT

### MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$10,000,000\*

7/76 - Continuing

**OBJECTIVES** – The Eastern Gas Shales Project (EGSP) was originated to establish a knowledge base and increase the production of natural gas from the Devonian Shale of the Appalachian, Illinois, and Michigan basins through advanced exploration and extraction techniques. According to calculations made by EGSP contractors, up to 1450 Tcf of gas may be in place in the Devonian Shales, while 280 Tcf are potentially recoverable. Technology R&D work is developing techniques for recovering this vast amount of gas. Successful project completion would elevate open flow potential of new shale gas wells from an average of 100 Mcf/d to as high as 350 Mcf/d and increase total gas reserves added per well from 300 to 600 MMcf.

**RECENT WORK AND ACCOMPLISHMENTS** – The three main areas of effort are resource characterization and assessment, technology research and development, and technology testing and verification. During FY 1977, \$4.2 million went toward research characterization and assessment, while \$5.8 million was spent on technology R&D and testing. In the area of resource characterization and assessment, the stratigraphic and structural geologic setting of the shale was defined regionally for the Illinois and Appalachian basins. A standard correlation of certain lithologic and stratigraphic units has been developed, isopach and structure contour maps of key black shale horizons in the Devonian sequence have been provided, data on structural characteristics have been compiled to determine orientation and extent of fracturing, and lineament maps have been prepared for certain states.

Devonian Shale cores were described for lithologic and fracture characteristics. Samples were collected for determination of index properties, directional characteristics, and flow properties. Samples were encapsulated at 10-ft core intervals at the well site to preserve down hole conditions. These encapsulated samples were tested for initial gas release volume and composition and were used to determine the kinetic relationships of volume and composition/time. An extensive series of elemental and mineralogical tests were performed on these samples to determine their relationship to gas release and production characteristics. The compatibility of specific fracture fluids with the Devonian Shales was investigated to determine which fluids will minimize formation damage. Five potential fracturing fluids were rated as to their relative effects on the swelling of shale samples.

In the area of technology research and development, field operations consisted of drilling, logging, and completing eight wells in four states. Gamma ray and differential temperature logs were used in delineating potential gas zones in the Devonian Shale. Seismic surveys were run in two specific areas. Fifteen zones in ten wells were treated using massive hydraulic (gelled water) fracturing, foam fracturing, chemical explosive fracturing, Keil fracturing, and cryogenic fracturing. Modeling studies of shale behavior in relation to fracturing have indicated the development of a secondary fracture surface not attached to the main fracture by a propped path. For future exploration use, production and oil and gas show maps have been made for almost all states in the three-basin area. In addition, maps of all wells into and penetrating the Devonian Shale have been constructed. Comparison of productivity from shale wells stimulated by borehole shooting tech-

\*Primarily pass-through funds for Eastern Gas Shales Project.

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niques and those stimulated by hydraulic fracturing techniques indicates that hydraulic fracturing will result in increased gas production (up to 60 percent higher). Proposed well site selections have been completed on six contracts and initiated on two additional contracts covering a total of 32 well sites.

In the area of technology testing and verification, a conference was held to solicit interest in cooperative government-industry exploration, drilling, stimulation, and completion activities in the Devonian Shales. Additional technology transfer to the private sector took place through workshops, a symposium, EGSP newsletters, and release of a color film on the EGSP.

**PLANS FOR THE COMING YEAR** – A major effort will be further development of the technology testing and verification aspect of the EGSP, in order to accelerate exploitation of the Devonian Shale gas resource. Work in this area will be integrated with the resource characterization and technology R&D portions of the EGSP in order to determine the most productive, economically and environmentally acceptable methods of recovering the Devonian Shale gas resource.

#### **GAS WELL FRACTURING IN THE DEVONIAN SHALE OF OHIO**

AMERICAN EXPLORATION COMPANY/VESCORP INDUSTRIES, INC.  
DOE - \$487,593; American Exploration/Vescorp Industries - \$517,842; Ohio ERDA - \$517,842  
12/10/76 - 1/10/79

**OBJECTIVES** – The three main objectives are to determine (1) the effectiveness of remote sensing imagery as a tool to optimize well locations in the Devonian Shale, (2) the relative effectiveness and quantitative benefit of foam fracturing and cryogenic fracturing to increase gas well production rates and reserves in the Devonian Shale, and (3) cost effectiveness of scaled-up versions of the better technique (foam or cryogenic fracturing) for improving gas well production rates and reserves in the Devonian Shale. An extensive geological study of Lawrence and Scioto counties in Ohio was to be conducted including analysis of available Landsat satellite and low- and high-altitude aircraft imagery to produce a fracture trace lineament map of the area and select nine well sites. The wells are to be drilled, logged, and cased, and in six of the wells, core will be taken from the Devonian Shale. Of these six, three will be stimulated with a foam fracture and the other three wells with a cryogenic fracture. The remaining three wells will be stimulated with scaled-up versions of the fracture treatment found to be most effective. The technology and cost effectiveness of the stimulation technique employed will then be determined.

**RECENT WORK AND ACCOMPLISHMENTS** – The geologic studies were completed, and six of the nine required sites selected. Drilling will commence as soon as a drilling rig can be subcontracted. The geologic report with site location recommendations was submitted in May 1977. Leasing and archaeological discoveries have reduced the number of usable sites to six. Alternate sites are being considered to develop the nine required sites.

**PLANS FOR THE COMING YEAR** – Final selection of the nine well sites will be completed. The wells will then be drilled, cored, and stimulated. Preliminary economic evaluations will be initiated after several months of on-line production in each well.

## PROJECT GASDEVEL—GAS PRODUCTION

COLUMBIA GAS SYSTEM SERVICE CORPORATION  
DOE - \$2,500,000; Columbia Gas System Service - \$2,303,715  
7/1/76 - 12/31/78

**OBJECTIVES** — The technical and economic feasibility of massive hydraulic fracturing (MHF) is to be demonstrated as an effective technique for increasing gas deliverability from the marginal gas reservoirs of the Appalachian Basin. Cryogenic and Kiel (dendritic) hydraulic fracturing techniques are also being developed and evaluated for possible use in stimulation of marginal gas resources in the eastern United States. The project also aims to use remote sensing imagery to identify lineaments and select 13 well sites based on the lineament densities. All 13 wells will be reservoir tested, production tested, and assessed to determine the technology and economic effectiveness of the various stimulation activities. MHF results will be analyzed, and in situ stress determinations and core analysis will be performed. All the preceding work is designed to provide technology and economic data for extraction of gas from a marginal resource.

**RECENT WORK AND ACCOMPLISHMENTS** — The status of well work is as follows. All well sites have been selected, and four of eight wells have been drilled, logged, and cased. Three of five wells have been cored. Five wells have been stimulated, one has been partially stimulated, and eight remain to be stimulated. The average production of the five wells prior to stimulation was 26.4 Mcf/d; average production following stimulation was 1089 Mcf/d. Well 20245 was plugged and abandoned, having produced no gas after stimulation. Well 20346, due to casing and tubing problems, was replaced. Production tests are currently underway to develop data for use in evaluating stimulation techniques. Data from conventional stimulation procedures are being assembled to help in the evaluation. A contract extension and modification is being negotiated and should be approved in the first quarter of FY 1978.

**PLANS FOR THE COMING YEAR** — During 1978, five wells will be drilled. Two of the five will be cored, and all five wells will be logged and cased. Nine wells will be stimulated in 1978. Four of them will be cryogenic fractured, three will be fractured using the Kiel process, and two wells will be stimulated by massive hydraulic fracturing. All production testing and evaluation work should be performed. Initial evaluations of all the techniques should be made if adequate data are available to determine the technology and cost effectiveness of the technique tested for improving production and reserves from the marginal gas resources located in the eastern United States.

## MASSIVE HYDRAULIC FRACTURING IN THE DEVONIAN SHALE

COLUMBIA GAS SYSTEM SERVICE CORPORATION  
DOE - \$2,154,155; Columbia Gas System Service - \$2,240,870  
6/18/75 - 12/31/77

**OBJECTIVES** — The stimulation program will assess the technical and economic feasibility of massive hydraulic fracturing (MHF) technology for the development of the Devonian Shale, an unconventional energy supply. Specific objectives are to select three well sites based on analysis of remote sensing imagery and geologic mapping, drill and core the wells, run wet and dry logs, and case the wells. For one well, four stages of formation stimulation will be designed and executed (at

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about 200- to 250-ft intervals) using about 200,000 gallons of fluid to hydraulically treat the entire shale section. In one well a maximum of four zones will be stimulated using 200,000 to 250,000 gallons of fluid. Reservoir tests will be conducted after each stage of stimulation for both wells. The remaining well will be designed for optimization of the target zone utilizing up to a maximum of four 250,000-gallon treatments. Reservoir test results will be obtained after the stimulation. An economic analysis of stimulating gas production using MHF will be performed. This development and testing of new stimulation techniques is desired by the Fossil Energy Branch of DOE to enable economic development and production of Devonian Shale gas resources.

**RECENT WORK AND ACCOMPLISHMENTS** – Drilling, coring, logging, and casing operations were completed on all three wells prior to FY 1977. During attempts to fill the first well to surface, hydrostatic pressure caused the exposed formation to fracture and take fluid. The same problem was experienced when cementing the casing in the well. The second and third wells were not filled to the surface for logging operations, and the casing was cemented in stages to reduce hydrostatic pressure and loss of fluids to the formation. Stimulation operations have been initiated on the first two wells, which were scheduled to be stimulated with an MHF in four separate zones. Four treatments were conducted using foam in Devonian Shale zones 1-4, Columbia Well 20403. One treatment was conducted using gelled water and sand in Zone 1 and two were conducted using a combination of gelled water and foam to emplace sand in zones 2 and 3, Columbia Well 20401. Pre- and post-fracture reservoir tests have been or are being conducted on all zones to help evaluate the effectiveness of the MHF. Laboratory tests have resulted in the following conclusions in regard to fracturing the Devonian Shale: (1) The Brown Shale Zone is the only Devonian Shale zone within which it is possible to contain a hydraulic fracture, (2) when fracturing multiple zones within the Devonian Shale, the Brown Shale must be fractured first, and (3) the bottom hole pressure while fracturing should not exceed the occurring stress in the Brown Shale by 600 psi or the fracture will not be contained in the Brown Shale Zone. Other laboratory studies on core samples have resulted in additional information: (1) Gas has been present in all core samples from the Devonian Shale, (2) the permeability of the shale is in the microdarcy range, (3) no free water was found in the core samples, (4) complete lithologic descriptions of all cores were made, and (5) other special core analysis studies were completed. Field tests have indicated that much quicker cleanup of the fracturing fluids occurs in foam fracturing than in the gelled water type fracture techniques. The cost effectiveness has not yet been determined for either foam or gelled water MHF treatments.

**PLANS FOR THE COMING YEAR** – The contract will be extended to allow all work to be completed. One more combination foam and gelled water MHF stimulation treatment is planned for Columbia Well 2041 in Devonian Shale Zone 4. Columbia Well No. 20403 and 20401 will be production tested for 6 months, and then all the testing results will be evaluated and MHF procedures will be optimized for stimulating Columbia Well 20402 in all four Devonian Shale zones. Upon completion of the MHF treatment on Columbia Well 20402, the results will be evaluated to determine the technical and economic feasibility of using MHF technology for the development of the gas resources of the Devonian Shale.



## DIRECTIONALLY DRILLED WELL IN DEVONIAN SHALE

CONSOLIDATED GAS SUPPLY CORPORATION  
DOE - \$582,356; Consolidated Gas Supply - \$168,935  
9/30/76 - 1/31/79

**OBJECTIVES** – This project was designed to demonstrate the technical and economic feasibility of using geophysical techniques, remote sensing imagery, near-surface stress measurements, surface joint measurements, and oriented core data to determine the optimum location and preferred orientation of natural fracture systems in the Devonian Shale formation. The project will also demonstrate the technical and economic feasibility of using directionally controlled, deviated well-bores stimulated with multiple-stage hydraulic fracture treatments to improve deliverabilities and reserves of wells drilled into the Devonian Shale. Secondary objectives include directionally drilling one well near Cottageville, West Virginia, logging the well, installing casing, and performing a multi-stage stimulation treatment in the Devonian Shale formation. Well tests will be conducted before and after the fracture treatment. Production will be monitored for 5 years and results reported. Results of the well completion will be evaluated to determine effectiveness of the exploration, drilling, and stimulation techniques.

**RECENT WORK AND ACCOMPLISHMENTS** – The geophysical field operations were conducted, and evaluation of the collected data is continuing. Remote sensing imagery work has been completed, resulting in the discovery of three dominant linear trends. Near-surface stress measurements resulted in dominant directions of lineation. The study of oriented core data was completed and resulted in three prominent trends of fracturing. Primary fracture systems in the core were vertical. These data will be evaluated to determine the direction required for the deviated well to intersect the greatest number of fracture systems. This is expected to enable development of a highly productive gas well.

**PLANS FOR THE COMING YEAR** – The geophysical operations will be completed and used to evaluate and determine the best site for the deviated well location. The remote sensing imagery, near-surface stress measurements, surface joint measurements, and oriented core data will be used for site selection. These data will also be used to determine the direction and possibly the angle of inclination required for the deviated well. The well will be drilled, logged, cased, tested, stimulated, and evaluated as noted in the objectives. The production monitoring task will be initiated. The available data will be evaluated to determine the technical and economic feasibility of using these techniques to improve deliverability and reserves of Devonian Shale wells.

## CHEMICAL EXPLOSIVE FRACTURING

PETROLEUM TECHNOLOGY CORPORATION  
DOE - \$751,806; Petroleum Technology - \$431,700  
7/1/76 - 9/30/78

**OBJECTIVES** – The technical and economic feasibility of chemical explosive fracturing in the tight lenticular Canyon Sands of the Val Verde-Kerr Basin in Texas will be demonstrated. Two new wells will be drilled in the Aldwell/Sawyer Field in Sutton County, Texas. Post-stimulation production will be compared with nearby producing wells.

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**RECENT WORK AND ACCOMPLISHMENTS** – Well W.E. Sawyer “4” No. 1, located some 20 miles southwest of Sonora was drilled with air and mist with a rotary rig to 7337 ft. Geological and engineering criteria were used for well location. Borehole audio trace surveys and differential temperature logs were run to identify zones with gas entry to well bore. Two intervals were identified. Pre-stimulation pressure buildup and flow tests were run. The well reached a maximum gas flow rate of 21.5 Mcf/d and a well head pressure of 1258 psig after 321-hour shut-in before stimulation. The well was shut in after pretesting to design high-temperature detonation equipment for the well.

**PLANS FOR THE COMING YEAR** – Final procedures to treat the drilled well are being prepared. The well will be treated with a chemical explosive frac after problems with equipment and early detonation at high temperature have been resolved. The well will be tested after stimulation. Depending on the results of stimulation in the first well, the second well in the program may be drilled, tested, and stimulated in the coming year.

#### **CHEMICAL EXPLOSIVE FRACTURING**

**PETROLEUM TECHNOLOGY CORPORATION**  
DOE - \$705,131; Petroleum Technology - \$249,001  
7/1/76 - 9/30/78

**OBJECTIVES** – The technical and economic feasibility of chemical explosive fracturing (CEF) will be demonstrated. CEF may be an effective stimulation technique for increasing gas deliverability from the unconventional energy supply, the Devonian Shales. Three wells are to be drilled in a 1258-acre parcel of land in Lincoln County, West Virginia. Production and flow test data will be used to compare the effectiveness of chemical explosive fracturing in this area.

**RECENT WORK AND ACCOMPLISHMENTS** – Geological and engineering criteria were used to select sites for the wells. The first well has been drilled and completed. This well was logged with gamma ray, compensated density, caliper, induction, and neutron tools. No measurable gas flow was encountered in the Devonian Shale prior to treatment. The well was treated with 29,400 lb of liquid explosives. However, due to malfunction in equipment, the explosive fracture did not proceed as planned. Subsequent investigation to find the cause of the problem created two down-hole gas-air explosions. Initial measurements after fracturing indicated that gas was flowing at the rate of 265 Mcf/d. After the secondary explosions, gas flow declined to 113 Mcf/d. The well is presently being monitored and producing between 18 to 66 Mcf/d against pipeline pressure of 20 psi.

**PLANS FOR THE COMING YEAR** – Two additional wells will be drilled and explosively fractured after detonator design problems have been eliminated.

#### **CHEMICAL EXPLOSIVE FRACTURING**

**PETROLEUM TECHNOLOGY CORPORATION**  
DOE - \$897,837; Petroleum Technology - \$436,963  
7/1/76 - 9/30/78

**OBJECTIVES** – The technical and economic feasibility of chemical explosive fracturing (CEF) will be demonstrated. CEF fracturing may be an effective stimulation technique for increasing gas

deliverability from the Devonian Shales. This work is to be done on one existing well and two newly drilled wells in Kentucky using liquid explosives of up to 30,000 lb. The two wells are to be drilled and completed and production tested prior to stimulating with chemical explosives, and one producing well is to be reworked and production tested prior to treatment.

**RECENT WORK AND ACCOMPLISHMENTS** – Well No. 685-1 in Perry County was chosen as the first test well on the basis of engineering and geological criteria. It was originally stimulated in 1965, with a hydraulic fracture, and produced about 60 Mcf/d. Gas production had declined to 9 Mcf/d. Before testing, time-consuming workover problems had to be overcome because of hole conditions. The gamma ray geophysical log indicated two potentially interesting zones. Temperature logs showed cooling effects of gas entry at two intervals in these “hot” zones. After workover operations were completed, gas flow was measured at 60 Mcf/d through a 2-inch Pitot tube. A 40-day pressure buildup test and a modified isochronal test were run prior to the current long-term production testing.

**PLANS FOR THE COMING YEAR** – After evaluation of two CEF stimulations on another Petroleum Technology Corp. contract in West Virginia, a decision will be made on the design of this well to permit simultaneous CEF treatment of all the indicated gas zones. Following this treatment, the well will be tested, and two new wells will be drilled to the Devonian Shale and treated with chemical explosives.

## CHEMICAL AND PHYSICAL ANALYSIS OF EASTERN SHALE

BATTELLE, COLUMBUS LABORATORIES  
DOE - \$499,800  
10/1/76 - 9/30/78

**OBJECTIVES** – The analysis and characterization of approximately 1000 Devonian Shale core samples will be done, maintaining the samples in their approximate “downhole” condition. After the characterization data for particular wells have been compiled, a multiple regression analysis will be employed to determine the interrelationships between the shale characteristics, the hydrocarbon gas content, and the well locations from which the samples were obtained. Shale characterization is essential to the Fossil Energy Technology Program’s R&D aspects of defining gas production potential. The characterization data complement other geological data to establish a characterization base. Correlation of this data base with stimulation and production data will indicate the variables that are related to increased gas production from the shale in specific geologic and geographic environments.

**RECENT WORK AND ACCOMPLISHMENTS** – A total of 379 samples, taken at 10 ft intervals, have been collected and encapsulated in pressure containers. These samples were obtained from 10 wells in the Appalachian and Illinois basins. The initial gas release analysis, both volume and composition, has been completed for all samples. The volume of gas per unit volume of shale ranged from 0.01 to 2.99, depending on the individual well and zone. All other types of analyses, both physical and chemical, have been initiated and are underway; for example, physical characterizations such as: density/porosity, apparent volume, and internal surface area measurements. Analysis of permeability and pore size distribution were performed only on selected samples where variability was expected. The gas release kinetics measurements, trace element analysis, SEM, XRD,

and petrographic work were also performed on selected samples. Preliminary correlations have been established using data compiled from the analytical work. A significant amount of scatter was observed in carbon content within the same well, and no correlation was found between carbon content and depth. The hydrogen content also showed data scatter with depth, but not to the degree indicated with carbon. Hydrogen/carbon ratios were unusually high in some of the wells. Indication of an inverse relationship was found between gas content and density and no relationship between density and porosity. A significant relationship was observed between carbon content and gas content.

**PLANS FOR THE COMING YEAR** — Work will include the completion of all samples previously collected and the sampling of new wells as they are cored. Analytical work will emphasize gas content and gas release kinetics and ultimate analysis on samples from new wells. X-ray diffraction, scanning electron microscopy, petrographic, elemental, and permeability analyses will be de-emphasized on new samples. An increased amount of statistical work will be performed in the coming year. Correlations will be made between the physical and chemical data already compiled and the stimulation and production data as it becomes available.

## EXPLOSIVE STIMULATION OF NATURAL GAS IN DEVONIAN SHALES

LOS ALAMOS SCIENTIFIC LABORATORY

DOE - \$350,000

3/1/77 - Continuing

**OBJECTIVES** — This work is designed to develop methods of stimulating the production of natural gas from wells in Devonian Shales by explosive permeability enhancement techniques.

**RECENT WORK AND ACCOMPLISHMENTS** — Successful prediction of explosive effects in geologic materials requires knowledge of the constitutive relations, or wave propagation and fracture characteristics, describing the material. Representative samples of Devonian Shales have been examined using gun- and explosive-driven shocks, as functions of bedding orientation and kerogen content. Hugoniot, ultrasonic elastic moduli, and time resolved behavior of the shock and refraction waves have been obtained, and the effects of these quantities on dynamic fracture are under investigation. These basic high-strain-rate rock mechanics studies are a continuing, long-term aspect of the program. Another aspect of the work involves development of heavy metal, explosively driven jet devices for downhole employment as a means of producing a series of horizontal pathways extending into the formation from a central borehole. These pathways serve as gas manifolding systems, intersecting existing natural fractures and effectively producing a larger-diameter borehole. They can also serve for liquid or slurry explosive emplacement or as stress concentrators for subsequent hydraulic fracture. A series of explosive experiments in limestone at the Nevada Test Site and in grout blocks which match Devonian Shale in density and compressive yield strength at Los Alamos have led to partial optimization of existing devices for maximum penetration. Penetration distances in excess of 28 charge diameters have been achieved using tapered liner charges of about 10-cm diameter. This penetration distance is now believed to scale with charge diameter, although more experimental data are required to confirm this. Typical hole diameters are 3-4 cm. These devices were originally developed under our weapons program, and have previously contained elements classified for national security. However, totally unclassified designs have now been developed which we believe will perform equally well for rock penetration applications. Development of these unclassified devices has been a major effort during the first few

months of the program. A computer based production/economic study, using a combined digital/analog computer system, is being conducted in conjunction with these efforts in order to evaluate the effectiveness of alternative stimulation procedures.

**PLANS FOR THE COMING YEAR** – Dynamic characterization of a greater variety of black and brown shales will be performed as the core samples are acquired. The explosive jet optimization effort, including both theoretical design and experimental verification, will be continued. Effects of compaction on permeability in the neighborhood of the jet path or explosive event will be studied. Permeability and SEM studies of this region will be performed and efforts made to overcome this effect through subsequent explosive treatment or chemical etching and leaching. More sophisticated computer studies of the reservoir drainage resulting from explosive stimulation in formations of varying permeability will be performed.

## LASER-INDUCED PYROLYSIS FOR CHARACTERIZATION OF DEVONIAN SHALES

LOS ALAMOS SCIENTIFIC LABORATORY

DOE - \$95,000

3/1/77 - Continuing

**OBJECTIVES** – This work is designed to develop methods that permit a rapid and reliable assessment of the gas and oil potential of specific sections of the Devonian Shale resource.

**RECENT WORK AND ACCOMPLISHMENTS** – Successful stimulation of gas flows from tight shale formations, such as the Devonian Shales, has proven difficult. Additional understanding of the nature of the source rocks, especially about the quantity and types of organic constituents in these geological formations is sought. Pulsed laser heating of discrete regions of core samples causes a rapid but controlled pyrolysis resulting in a series of hydrocarbon and other gaseous products. Analysis of the type and quantity of these products is being used to rapidly assay samples of shales. Emphasis during this period has been on the construction of equipment that will serve as prototypes for eventual field utilization. Sampling techniques have been developed that lead to acceptable precision and reliability. These results show that adequate quantities of a wide variety of gaseous products do result from laser spot heating.

**PLANS FOR THE COMING YEAR** – Characterization studies will be expanded to include three general areas: (1) Gas (and oil) yields—Methods will be established to utilize this technique for the routine analysis of shale samples with a single, rapid measurement. Precision and accuracy will be defined. Equipment necessary to conduct similar tests at a field coring site (to analyze freshly exposed cores or drilling chips) will be designed, constructed, and tested. (2) Detailed rock analysis—Concurrently, work will commence on the characterization of large molecular fragments

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## PHYSICAL AND CHEMICAL CHARACTERISTICS OF DEVONIAN SHALE SAMPLES

MOUND LABORATORY

DOE - \$369,000

10/1/76 - Continuing

**OBJECTIVES** – Samples of the Devonian Shale are to be analyzed and characterized in their approximate “downhole” condition. These characterizations include determination of fuel yield and fuel characteristics, a detailed characterization of the kerogen, the relationship of hydrocarbon release to mechanical loading, and spectroscopic and microscopic physiochemical characterization. The production of gas from unconventional sources, such as the Devonian Shale, is an integral part of the Fossil Energy Technology Program. The development of a data base by physical and chemical characterization, to complement the data obtained from drilling logs and geological investigation, is an important part of the R&D effort on Resource Characterization. This data base can be used to establish relationships between Resource Characterization and stimulation and production data to determine the controlling variables in different geographic areas and geologic environments.

**RECENT WORK AND ACCOMPLISHMENTS** – All of the geochemical tasks are nearly on schedule. Organic carbon has been completed on all samples; kerogen analysis and vitrinite reflectance have been completed on 90 percent of the samples. Fischer Assay tests have been completed on four wells (of eight sampled), and the additional four wells are in progress. Mound Laboratory has initiated special studies of moisture content, clay mineralogy, and dilatometry of selected samples. Also added to the contract were biostratigraphic studies of all cores, a statistical analysis of the Clinton Sand, and archeological surveys of potential well sites in Ohio. Initial statistical correlation analysis shows that a significant correlation exists between the total hydrogen content, oil yield, organic carbon content, and total gas content in the Martin County, Kentucky, well. Other correlation analyses and analysis of additional wells are in progress at this time.

**PLANS FOR THE COMING YEAR** – The original suite of analyses for all samples received to date, which were taken at 30-ft intervals, will be completed. Plans to sample at a closer interval during the coming year will be implemented. All the additional special studies will be continued. Mineralogical and elemental analysis will also be performed in addition to the original analyses on all future samples. It is anticipated that as the analysis of the sample is completed, additional statistical analyses will be performed. At this time, correlations between characterization and stimulation/production data will be attempted.

## APPRAISAL OF DEVONIAN BLACK SHALE IN THE APPALACHIAN BASIN

UNITED STATES GEOLOGICAL SURVEY

DOE - \$1,859,480

1/1/76 - Continuing

**OBJECTIVES** – This interagency agreement is designed to provide a high level of expertise to work with and coordinate other contractors characterizing Devonian Shale in the Appalachian Basin. Specific objectives include structural and stratigraphic analysis and correlation, assessment of shale thermal maturity as a gas exploration tool, and compilation and encoding of all EGSP data in a project ADP System. The overall coordinating role of the USGS will allow all contractor data for the Appalachian Basin to be presented to the public as quickly as possible. This will permit

exploration rationale to be developed and tested in conjunction with the fracture stimulation testing currently ongoing as part of the evaluation of this unconventional energy supply. All data coordinated by the USGS will be used by DOE to describe the gas resource potential associated with Devonian Shale.

**RECENT WORK AND ACCOMPLISHMENTS** — USGS tasks are divided into several work areas. The structure and stratigraphy work intends to map lineaments in Ohio and West Virginia from remote sensing imagery, to coordinate stratigraphic and structural work being done by other contractors, to map joint and fracture features in several areas of the Appalachian Basin, to prepare isopach and structure contour maps of two principal black shale units and to prepare six regional cross sections coordinating subsurface correlation of units throughout the basin. The clay mineralogy work involves establishment of a disaggregation technique for the shale samples. Thermal maturation studies of the shale concerning measurement of vitrinite reflectance and conodont color index (CCI) show a good preliminary correlation to gas content in the shales. Two papers discussing these results have been prepared. Other geochemical work involving analysis of C,S,N isotopes, uranium, and other trace elements has begun on several cores from areas in the Illinois and Appalachian Basins. The borehole gravimeter geophysical studies indicate the tool will not be useful as a method of evaluating fracture porosity and density in the Devonian Shales. Work on three wells indicates there may be a determinable correlation between shale organic content and density and gamma ray curves. The EGSP ADP System is being coordinated by Petroleum Information Corporation, a sub-contractor to USGS. As of the end of FY 77, the system was not operational. The USGS resource appraisal work is not scheduled to begin until FY 79.

**PLANS FOR THE COMING YEAR** — USGS coordinating activities will continue for the Appalachian Basin. Several workshops will be held to assist flow and discussion of ideas among all pertinent contractors. Field geologic work to obtain additional stratigraphic and structural data will continue. Clay mineralogy, uranium and trace element, and conodont analyses will be performed on core samples submitted from sites primarily in the Appalachian Basin. The ADP System will be initiated as a storage and data manipulation facility available to all contractors. The geophysical work will be modified to evaluate potential use in relation to organic content determination.

## CHARACTERIZATION OF EASTERN U.S. DEVONIAN BLACK SHALE

ALFRED UNIVERSITY  
DOE - \$218,889  
10/1/76 - 1/31/78

**OBJECTIVES** — The contractor will perform elemental tasks which are part of the shale characterization. Shale core from the three Eastern basins will be analyzed to determine the bulk mineralogy, clay mineralogy, organic geochemistry, presence of trace elements and physical and geochemical properties of the shales. Appropriate charts, maps, and diagrams will be constructed to illustrate the results of these studies. This project is specifically directed toward determining the magnitude of potential natural gas reserves and increasing production in the three Eastern shale basins. The shale will be thoroughly characterized by the contractor in the areas of geochemistry and physical properties. The analytical studies for the characterization of Eastern hydrocarbon-bearing shales will include detailed studies on both outcrop and subsurface samples. Parts of the analytical studies will be made through contracts and/or cooperative agreements with industry,

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educational institutions, and other governmental agencies. The contractor will (1) classify and identify the Eastern shale deposits with respect to their chemical, physical, and mineralogical properties; (2) obtain information to aid in well-log interpretation, i.e., bulk density logs, kerogen logs, fracture porosity, and radioactivity logs, to aid in a more rapid field analysis of the resource; (3) supply information to aid in recovery of reserves, through well-stimulation techniques such as fracture design; and (4) evaluate techniques of analytical instrumentation for a rapid resource assessment.

**RECENT WORK AND ACCOMPLISHMENTS** – Several analytical and testing procedures were developed and additional data were obtained on the characterization of the practice samples of the Devonian Black Shales. Analytical procedures for boron, fluorine, chlorine, and bromine have been worked out and adopted. It was found that several elements could not be determined without a plasma spectrograph. Procedures have been worked out to identify the presence or absence of the clay minerals, montmorillonite and kaolinite. Neither of these minerals were found in the practice samples from the Upper Devonian Dunkirk and Middlesex and the Middle Devonian Chittenango shale. A spray drying procedure was developed for the preparation of randomly oriented samples for quantitative X-ray diffraction analysis. In the physical testing laboratory, the procedures for density, porosity, and strengths have been completed. New data for 13 elements in three Devonian Shales are reported, and density measurements on several samples are available.

**PLANS FOR THE COMING YEAR** – This contract ended on September 30, 1977, with incomplete results. An agreement was reached between Alfred University and DOE whereby Alfred University would complete the scope of work under a no cost extension.

## ENERGY RESOURCES OF DEVONIAN SHALE IN THE APPALACHIAN BASIN

UNIVERSITY OF CINCINNATI  
DOE - \$183,513  
10/1/76 - Continuing

**OBJECTIVES** – Special geochemical and petrologic studies of the Devonian Shale will be done to complement the geological studies of, and prepare a depositional model for, the entire Appalachian Basin. The production of gas from an unconventional source such as the Devonian Shale is an integral part of the Fossil Energy Technology Program, and Resource Characterization is one of the major tools which can be used to locate potential geologic horizons and geographical areas where the chance of success is greatest. The correlation of geological, physical and chemical variables with production and stimulation technology can lead to the right technology choice for a given horizon and area.

**RECENT WORK AND ACCOMPLISHMENTS** – The geological evaluation work, preparation of a depositional model for the Appalachian Basin, has been centered around the analysis of paleocurrents in the outcrop belt. Paleocurrent directions have been plotted in western New York, northwestern Pennsylvania, northeastern Ohio, and southeastern West Virginia and combined with data from a previous study in eastern New York, central Pennsylvania, and northeastern West Virginia. These data with an interpretation of current flow were presented in a paper published at the EGSP Symposium. A useful stratigraphic marker, the Three Lick Bed, was mapped from southern Ohio and West Virginia to northwestern Tennessee. These data were presented in a publi-



cation designated as MERC/CR-77-2. A comprehensive annotated and illustrated bibliography of argillaceous sediments was published as MERC/CR-77/8. The geochemical characterization work has consisted of carbon and carbon isotope studies and the completion of a study of exchangeable cation in the shale. The exchangeable cation paper is in preparation at this time.

**PLANS FOR THE COMING YEAR** – The final contribution to the basin-wide stratigraphic analysis, maps of paleocurrent analysis, and preliminary maps of geochemical variables are to be completed. A definitive analysis of SEM use for paleocurrent studies, V/Ni analysis for all samples, and fabric type definition in the shales are also to be completed.

## **THE NEW ALBANY GROUP AS A SOURCE OF HYDROCARBONS**

ILLINOIS STATE GEOLOGICAL SURVEY

DOE - \$448,476

10/1/76 - Continuing

**OBJECTIVES** – The DOE sponsored program is a geologic and geochemical study of the New Albany Group (Devonian Black Shale) in Illinois relative to its potential as a hydrocarbon source, particularly natural gas. The program is keyed to the national goal of developing a greater self-sufficiency in energy resources in the United States in the next 10 years. Devonian Shale formations in the Appalachian Basin, from eastern Tennessee to southern New York, have yielded significant quantities of natural gas. In the Big Sandy gas field of southeastern Kentucky, more than 2 Tcf of gas has been produced from about 200 cubic miles of gas-bearing black shale. This is a production rate of about 1 ft<sup>3</sup> of gas per ton of shale, using the present method of well stimulation and production. Laboratory tests of the black shale show that by pyrolysis the yield of gas can be increased by as much as 3 orders of magnitude. Thus, the Devonian Black Shale of the Appalachian, Michigan, and Illinois basins constitutes a vast potential reservoir of natural gas that might be utilized if suitable extraction techniques could be developed.

**RECENT WORK AND ACCOMPLISHMENTS** – Six cores (five from Illinois and one from western Kentucky) provide the principal data base for these investigations. Four of these have been obtained through DOE, the other two from USGS files. Using subsurface stratigraphic techniques, 22 cross sections and a preliminary New Albany Group thickness map have been prepared. In addition, isopach maps are in preparation for each of the formations within the New Albany. Black shales predominate in the center of the Illinois Basin, whereas gray shales predominate around the margins of the Basin. A computer data base of over 5000 wells penetrating the New Albany Group has been compiled. LANDSAT photo studies of western Illinois surface linear features suggest that most of these linears are of glacial origin. Tectonic origins are not postulated for any of the linears, and caution is advised when interpreting linear features in glaciated terrains.

Mineralogic and petrographic studies have concentrated on two cores. A preliminary classification of four shales lithofacies based on primary depositional characteristics has been established and related to oxygenation of the bottom environment. Preliminary results of physical index properties; major, minor, and trace elements; and gas compositions have been obtained for three cores. Internal surface area measurements reveal that gray shales have higher ultramicroporosity than do black shales. The gray shales also have high gas diffusion rates and are better reservoir rocks.

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**ANS FOR THE COMING YEAR** – The process of data collection and organization will be coordinated and the results in the DOE Eastern Gas Shales Project in the Illinois Basin will be evaluated. Specific steps in this process will include: (1) compiling available drill hole and outcrop data on the New Albany Group in Illinois, (2) studying the stratigraphy and structure of the New Albany Group, along with overlying and underlying rocks in Illinois, to determine relationships to occurrence of hydrocarbons, especially natural gas, (3) obtaining selected data for comparison with geophysical logs of the New Albany Group, (4) characterizing in detail the New Albany Group in Illinois with respect to physical, mineralogical, and chemical properties and hydrocarbon content by detailed analyses of core material to delineate targets that would be the most favorable for production of hydrocarbons under more sophisticated techniques of extraction, (5) encoding all geologic and chemical data obtained in the investigation in a computer format, and (6) characterizing geologically the Eastern Shales to aid in evaluating their economic importance.

### STUDY OF THE NEW ALBANY SHALE IN INDIANA

INDIANA DEPARTMENT OF NATURAL RESOURCES GEOLOGICAL SURVEY

DOE - \$233,360

10/1/76 - Continuing

**OBJECTIVES** – This contract will determine the geological, geochemical, and physical characteristics of the Devonian Shales in Indiana, in order to delineate target areas where commercial reserves of natural gas are likely to be present. Specifically, all available drill hole and outcrop data will be analyzed for the Devonian Shales in Indiana, the stratigraphic and structural framework of the shales will be studied and the potential resource of shale gas in Indiana as well as the most efficient method of extracting it will be determined.

**PRESENT WORK AND ACCOMPLISHMENTS** – Work accomplished to date by the Indiana Survey includes a complete regional stratigraphic cross-section net for the State of Indiana in the Illinois Basin. This net consists of eleven cross sections, using 109 geophysical well logs as control points. Three additional cross sections have been constructed for the southern portion of the Michigan Basin. A structure contour map on the base of the New Albany Shale and its equivalents was compiled from about 1,400 well logs in the Illinois Basin and 400 well logs in the Michigan Basin. A lithologic map of the New Albany Shale and equivalent strata in Indiana has been constructed, as well as a map of gas fields and gas shows in the same area in the New Albany and in the Middle Devonian limestone formations. Additional work has involved the mapping of fracture and lineament orientations for use in future exploration for fractured reservoirs.

**ANS FOR THE COMING YEAR** – Plans include the construction of specific horizon structure maps and lithofacies maps, continuation of the fracture and lineament orientation studies, and intensive geochemical and physical testing of Devonian Shale core material.

### STUDY OF HYDROCARBON SHALE INTERACTION

JUNIATA COLLEGE

DOE - \$45,000

10/1/76 - Continuing

**OBJECTIVES** – The two main objectives are the determination of the adsorption capacity and permeability of the shale and the degassing rates of hydrocarbons as a function of foreign gases. The

critical parameters measured are methane and helium isotherms, BET surface areas, and diffusion constants for methane through shale. These measurements are necessary for estimating well production from the known fracture system and wellhead pressure. In addition, the data should determine whether the "flooding effects" of foreign gases will release hydrocarbons.

**RECENT WORK AND ACCOMPLISHMENTS** – The first steps toward the construction of an automated diffusion/isotherm system for simultaneous operation via multi-tasking Fortran on a computer were completed. This first system is an automated apparatus for sub-atmospheric work with temperature control, instant determination of zero time, and unattended running for several hours. A second system will have the same capabilities as the first but at pressures up to 500 psi and possibly higher. The possibilities for gas present in shale are (a) open porosity, (2) adsorption, and (3) solution in kerogen and water. Sorption isotherms were determined as a function of gas and temperature on samples from Well 20403. The data indicated that gases tend to be sorbed by shale in an increasing order of their molecular polarizability and/or boiling points. The open porosity model must be rejected as applied to hydrocarbon gases in shale. The concept of specific degasibility has been developed. The two rock parameters in general use are porosity and permeability. However, high porosity may have low permeability and vice versa. A single combined parameter to assess a rock's potential productivity would be most useful. Specific degasibility is a single parameter which is a measure of the gas given off for a given pressure change and area of rock exposed. This parameter combined with an estimate of the magnitude of a fracture system and the initial pressure of methane in the rock allows estimates of the amount of gas a given well will produce. Specific degasibilities, helium densities and amounts of non-gaseous methane residing in the well samples were calculated. An average degasibility for Well 20338 was about  $8.2 \times 10^{-2}$  cc gas/cm<sup>2</sup> torr sec<sup>1/2</sup> while for Well 20336 it was  $2.9 \times 10^{-7}$  cc gas/cm<sup>2</sup> torr sec<sup>1/2</sup>. Given the same initial wellhead pressure and comparable fracture systems, Well 20338 should be 3 times more productive than Well 20336. Most of the methane was found to be in a non-gaseous state (adsorbed or in solution). Well 20336 had 77 percent in a non-gaseous form, while Well 20338 had 88 percent non-gaseous methane. The effects of foreign gases on hydrocarbons degassed were tested on 20 samples of shale from Well 20403. Samples were subjected to atmospheres of CO<sub>2</sub>, H<sub>2</sub>S, O<sub>2</sub>, and H<sub>2</sub>O. Nonpolar gases, O<sub>2</sub> and CO<sub>2</sub>, showed no evidence of hydrocarbon gas displacement. H<sub>2</sub>O showed some H<sub>2</sub>, H<sub>2</sub>S, or CO<sub>2</sub> production in some cans. H<sub>2</sub>S was sorbed by the shale in all cases and a significant number of cans showed a production of hydrocarbon gas.

**PLANS FOR THE COMING YEAR** – Immediate plans are the completion of software for completely automated isotherm/diffusion determinations. The program for the isotherms has been completed as of November 1977. Work continues on the program for diffusion constant determinations. The software will be set up on tape and be available for the new computer when it arrives sometime in December 1977. There is a backlog of about 100 samples to be completed for Well 20403. New samples from planned wells are expected to arrive beginning in January 1978. In the spring of 1978 the construction of a second apparatus for high pressures will begin. By next summer, two complete apparatuses will be giving data to a central computer system. More experimental work will be accomplished on the diffusion constants for slabs as a function of pretreatment of the surface of a slab with particular reference to the effects of water.

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## BLACK SHALES OF APPALACHIAN BASIN AND KENTUCKY

UNIVERSITY OF KENTUCKY RESEARCH FOUNDATION

DOE - \$270,626; University of Kentucky - \$20,009

10/1/76 - Continuing

**OBJECTIVES** – The study will determine the geological, geochemical and physical characteristics of the Devonian Shales in eastern Kentucky, the most productive shale gas producers. Specifically, this work will result in the development of a computerized data base to allow comparison of geologic and gas production data, and to allow statistical analysis of contract-generated geochemical data. Additionally, stratigraphic studies will be correlated with lithologic, physical, and gas production data, and the geologic history of the Upper Devonian-Lower Mississippian Age rocks in the study area will be determined. This detailed study will provide the data required to assess the Devonian Shale gas resource and provide input for determining recoverable gas reserves and developing cost-effective extraction technologies.

**RECENT WORK AND ACCOMPLISHMENTS** – Well-log inventories from over 1500 wells have been assembled, preparatory to generating structure contour and isopach maps. All of the stratigraphic sections along the eastern Cincinnati Arch outcrop belt have been measured and described. Sixty shale samples from the outcrop belt have been analyzed for 25 major, minor, and trace elements. Four regional cross sections and a tie section have been completed and submitted to the USGS. The data bank, which will tie all parts of the study together, is being set up with much of the work already initiated.

**PLANS FOR THE COMING YEAR** – All contracted work will continue; finishing the well-log inventory and bringing the data bank on-line will be emphasized. Stratigraphic sections will be measured along the Pine Mountain overthrust belt, geochemical work will determine which elements, by virtue of their stratigraphic and areal variation, carry the greatest amount of geochemical information.

## APPRAISAL OF DEVONIAN BLACK SHALES IN NEW YORK STATE

NEW YORK GEOLOGICAL SURVEY

DOE - \$59,475

10/1/76 - Continuing

**OBJECTIVES** – Work under this contract will identify the structural framework and production potential of the Devonian Black Shales in New York State. Specifically, the stratigraphic and lithologic units in the Black Shale in New York and adjacent states will be defined, possible shale gas development areas will be identified, and structure contour, isopach, and lithofacies maps will be prepared. Data from field studies and well logs will provide the base to accomplish these objectives. These data will complement geologic, geochemical, and physical characterization data provided by other contractors in the state and will provide the necessary information for use in exploration for Devonian Shale gas.

**RECENT WORK AND ACCOMPLISHMENTS** – The contract work has been divided into three major areas: stratigraphy and structure, exploration, and data handling and encoding. Preliminary versions of six stratigraphic cross sections covering New York, tied in also with Pennsylvania, have been completed to illustrate the subsurface geology of the Devonian Shales. Using these and other

geologic maps, regional stratigraphic correlations were made. Isopach and structure contour maps of two Devonian Shale units found in New York were completed. Source data for lineament maps were compiled from remote sensing imagery. Base maps for the maps of Devonian Shale wells, and current and past oil and gas production, have been prepared. Prime target gas exploration areas were identified based partly on structure data and knowledge of prior production from specific areas and identifiable geologic horizons.

**PLANS FOR THE COMING YEAR** – Structural correlations and final versions of cross sections, lithofacies and structure contour maps, and well cuttings analyses will be completed in FY 1978. All production data and well locations will be compiled on overlays of the base maps for final maps of these items. Data encoding will begin, as information from about 1300 wells is expected to be put in the system. By the end of FY 1978, the New York Geological Survey in conjunction with DOE will be providing additional information to the public that will be useful for evaluating the potential of the Devonian Shales.

### TIOGA BENTONITE, A MARKER HORIZON IN THE DEVONIAN SHALE

UNIVERSITY OF NORTH CAROLINA

DOE - \$12,495

10/1/76 - 3/31/78

**OBJECTIVES** – The Tioga Bentonite is a thin unit containing beds of ancient volcanic ash deposited during a brief interval of time throughout most of the Appalachian Basin. It is variously positioned relative to the base of the Devonian shale, potentially a major resource of natural gas in the East. Since the Tioga Bentonite is a very precise isochronal marker (plane of equal time) penetrating the shale, it affords an opportunity to better understand the internal stratigraphy of the shale, knowledge of which is essential to efficient exploration of the resource. The purpose of this research is to study the stratigraphy of the Tioga Bentonite and its relationship to the Devonian Shale and to characterize the bentonite petrographically.

**RECENT WORK AND ACCOMPLISHMENTS** – The Tioga Bentonite has been documented in over 400 well sites and 100 outcrops. These localities have all been verified; i.e., the positions relative to geographic coordinates have been located and information on stratigraphic position, thickness, and lithologic characteristics plotted according to a standardized data format. Maps and cross sections are being prepared showing (1) control points, (2) total thickness of the Tioga interval, (3) total thickness of volcanigenic strata (excluding shale interbeds), (4) thickness of the middle coarse marker bed within the Tioga interval, (5) number of recognizable ash beds, (6) details of the internal Tioga stratigraphy, (7) relationships of the Tioga Bentonite to directly underlying or overlying stratigraphic units, and (8) maximum diameter of five key minerals from the middle coarse marker bed as viewed through a petrographic microscope. These are now complete or nearing completion. Petrographic characterization of the Tioga Bentonite is also complete and that section of the final technical report has been written. The remainder of the report is currently in progress. The principal investigators presented a paper summarizing the results of their research at the First Eastern Gas Shales Symposium on October 18, 1977.

**PLANS FOR THE COMING YEAR** – Submission of the final technical report and the maps and cross sections listed above is anticipated January 1, 1978.

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## STUDY OF UPPER DEVONIAN SHALE IN OHIO

OHIO DEPARTMENT OF NATURAL RESOURCES, GEOLOGICAL SURVEY  
DOE - \$170,894  
10/1/76 - Continuing

**OBJECTIVES** – Four types of studies will be performed. The first is to collect, compile, and analyze all available well, core, and outcrop data on the Devonian Shale in Ohio. The second is to study the stratigraphic and structural framework of the Devonian Shales and associated lithologic units above, below, and laterally equivalent to the shale interval. The third is to perform lithologic and geochemical analysis of the core material. The final objective is to encode all data in a prescribed format. Completion of these objectives will enable investigators to determine areas where commercial volumes of gas are likely to succeed. This work forms an integral part of the R&D effort in the establishment of a Resource Characterization data base to determine the relationship between geologic, physical, and chemical variables to production/stimulation data generated during the project. By identifying geological horizons or geographical areas with a high success of gas production from an unconventional source such as the Devonian Shale, the project becomes an important part of the Fossil Energy Technology Program.

**RECENT WORK AND ACCOMPLISHMENTS** – The Devonian Shale well map and Ohio gas production map were completed early in the contract period. Preliminary isopach maps of the radioactive facies of Lower Huron Member, Upper Huron Member, and the Rhinestreet equivalent in Ohio were initiated and completed during the final quarter. A preliminary structure map on the base of the Lower Huron was also completed and submitted to the USGS for incorporation into the regional structure map. Samples were collected, representative splits were removed, and preparation initiated for the physical and chemical characterization tasks. Lineament definition from remote sensing data was initiated using LANDSAT multiseasonal transparencies.

**PLANS FOR THE COMING YEAR** – The Ohio Geological Survey plans to construct a series of interlocking stratigraphic cross sections and a series of isopach and lithofacies maps of the Devonian Shale and of distinct lithologic units within the shale. A series of structure contour maps are planned on units above and below the Devonian Shale as well as on units within the shale. Density and grain size analysis is planned in the physical characterization area. Geochemical characterization work planned includes X-ray diffraction, thin section petrography, and SEM for the mineralogical analysis. Chemical analysis will include differential thermal analysis, elemental analysis, proximate analysis, and ultimate analysis. In the resource assessment area, work planned includes the monitoring of gas shows and correlation of gas shows with lithologic and geophysical log data. Additional work is planned in the area of lineament delineation by remote sensing utilizing LANDSAT, Skylab, and ERTS imagery. Work is also planned to initiate encoding data into a prescribed ADP System.

## STRATIGRAPHY OF PENNSYLVANIA DEVONIAN BLACK SHALES AND RELATED ROCKS

PENNSYLVANIA TOPOGRAPHIC AND GEOLOGIC SURVEY  
DOE - \$81,073  
10/1/76 - Continuing

**OBJECTIVES** – Work under this contract includes compilation and presentation of available data on the Devonian Shales in a form suitable for exploration evaluation of target sites. Specific

objectives include preparation of structure contour, isopach, and lithofacies maps, cross sections, a drilling depth map and maps illustrating historic production, gas shows and all wells penetrating the Devonian Shale. Identifying prime target areas for Devonian Shale gas development based on geology is an integral part of DOE's R&D effort in evaluating the potential associated with this unconventional fossil energy source.

**RECENT WORK AND ACCOMPLISHMENTS** — The contract work has been separated into three major areas: structure and stratigraphy, production, and data encoding. The first two tasks are on schedule and the third is scheduled for FY 1978. Local and basinwide cross sections and structure contour maps on two of the black shale units within the Devonian Shale have been completed and are ready for publication. Data have been compiled and are continuing on the lithofacies maps. Production data in the form of maps for gas shows, Devonian Shale wells and overburden depth in Pennsylvania have been prepared in preliminary form and are about 95 percent complete. Data compilation has been ongoing in conjunction with the above activities.

**PLANS FOR THE COMING YEAR** — Structure and stratigraphy work will be ongoing in FY 1978 with completion of additional structure contour and isopach maps for the state. Data encoding is to be completed by the end of FY 1978 in order that this information can be evaluated and correlated throughout the basin.

#### **EVALUATION OF CHATTANOOGA SHALE IN THE TENNESSEE VALLEY AND RIDGE**

TENNESSEE DEPARTMENT OF CONSERVATION  
DOE - \$209,971; Tennessee Department of Conservation - \$5,396  
10/1/76 - Continuing

**OBJECTIVES** — This study will document the regional structure and stratigraphy in the Tennessee Valley and Ridge, locate the Chattanooga members which contain high hydrocarbon values; identify by seismic profiles the location of tectonically fractured zones in the Chattanooga shale; and evaluate the potential of the shale for natural gas and uranium. The Chattanooga shale is 400 to 2,000 ft thick in the Valley and Ridge Province of eastern Tennessee and a potentially significant source of natural gas. The shale in the area has been subjected to intense tectonic activity that may have created fracture zones which persist beneath several of the major Valley and Ridge thrust sheets. These fracture zones would constitute potential gas reservoirs. Successful location and tapping of these reservoirs would contribute to the domestic energy base.

**RECENT WORK AND ACCOMPLISHMENTS** — Bids were received for subcontract work from three companies to do field work and processing of a seismic survey, three seismic refraction studies and a comprehensive report in the Valley and Ridge. The survey consisted of two seismic lines totaling 90 miles which were run normal to the structural trends in eastern Tennessee. A subcontract was awarded to Geophysical Services, Inc. (GSI) in Houston, Texas, January 1977. GSI commenced field work in February 1977 without a signed subcontract and completed the survey in March. The subcontract was approved and signed on March 10, 1977. The Division of Geology (Tennessee) made a reconnaissance of the routes to be traversed and provided personnel to escort the seismic crew. Geologic information, from published and unpublished geologic maps and strike and dip measurements of outcrops, was compiled along the routes of the seismic lines, and composite strip maps were made by the Division for GSI's use in interpreting the seismic data. GSI processed the seismic data and began interpretation and preparation of their report to the Division



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of Geology which reviewed their work as it progressed and received some of the preliminary seismic sections. Locations were selected for obtaining oriented cores in the Chattanooga shale. The two areas were selected in synclinal troughs, where the formation appeared to be relatively flatlying. Structural cross sections along and across the southwestern margin of the Pine Mountain block were drawn, based on surface geology and available well data. The cross sections were prepared as part of a collateral study supported by the USGS.

**PLANS FOR THE COMING YEAR** – Delays in starting the subcontract seismic program have set the project approximately one quarter behind schedule. Core drilling and stratigraphic studies were shifted to the second year of the project. The interpretation of the seismic data should be completed and delivered by December 1977. Identification of tectonically fractured zones will be made when the seismic data are made available. Sites will be selected for taking two 4-inch-diameter oriented cores, one on Newman Ridge and the other on Clinch Mountain. Five coring locations will be selected along Newman Ridge and Clinch Mountain for obtaining NX cores through the Chattanooga shale near its outcrop. Descriptive lithologic logs of the oriented cores and NX cores will be prepared. Based on the results of the core drilling, a stratigraphic study of the Chattanooga shale will be completed and stratigraphic cross sections prepared. The evaluation of the resource potential of the Chattanooga shale in the Tennessee Valley and Ridge will be completed.

## EVALUATION OF DEVONIAN SHALES IN WEST VIRGINIA

WEST VIRGINIA GEOLOGICAL SURVEY  
DOE - \$660,364; West Virginia Geological Survey - \$123,390  
10/1/76 - Continuing

**OBJECTIVES** – This work will characterize the geochemical, geological, and physical properties of the Devonian Shales in West Virginia and evaluate the shale sequence to determine the potential gas resource contained in the shales. The specific objectives of this work are to analyze all available drillhole and outcrop data on the Devonian Shales in West Virginia and conduct a comprehensive study of the sedimentary history and stratigraphic framework of the shales and closely associated lithologic units. These studies will delineate the geologic relationships between various lithologic units and how these relationships bear upon the occurrence of natural gas. This work will result in the delineation of target areas where commercial volumes of shale gas are likely to be present and yield information as to what production stimulation techniques would be most likely to succeed.

**RECENT WORK AND ACCOMPLISHMENTS** – All scheduled tasks have been initiated. Work towards meeting the objectives outlined above has included the construction of base, structure contour, and isopach maps, 209 X-ray diffraction analyses for mineral determination, radiography of 15 shale samples, development of a regional stratigraphic framework for West Virginia and completion of five stratigraphic cross sections.

**PLANS FOR THE COMING YEAR** – Work will continue on the initiated tasks as core material becomes available and mapped coverage of West Virginia is expanded. The delineation of target areas is not scheduled to begin until FY 1981.



## GEOLOGICAL ANALYSIS OF GAS PRODUCTION FROM DEVONIAN SHALE IN THE APPALACHIAN BASIN

WEST VIRGINIA UNIVERSITY  
DOE - \$164,232  
10/1/76 - Continuing

**OBJECTIVES** – The program will identify and determine the relative importance of structural parameters influencing the production of gas from an unconventional energy source, the Devonian Shales. Specific objectives have been to construct structure contour maps of eastern Kentucky and West Virginia, to determine if types, styles, and outcrop manifestations of structure reflect production characteristics, to determine whether near-surface fractured and faulted zones, if detected by shallow seismic surveys, can be related to shale production and remote sensing lineament data, and to determine if groundwater flow and gas productivity are correlatable in areas of gas production. This detailed study of structural features has potential for developing exploration techniques to determine prime target areas for extraction of Devonian Shale gas. This gas source is one of four unconventional sources currently being investigated by the Fossil Energy Branch of DOE. Successful techniques for exploration used in production of this gas are needed to complement development of fracture stimulation methods being conducted by other contractors.

**RECENT WORK AND ACCOMPLISHMENTS** – The contractor has separated work into five major areas: regional structure, production, groundwater, field structure, and geophysics. Overall, the progress and accomplishments have been satisfactory in the first three areas but delays have occurred in the last two. Structure contour maps on five horizons have been completed for all or segments of 38 quadrangle maps. Data for this study were taken from well records, geologic maps, and other published sources. These data will be useful on a regional basis to all contractors working in the Appalachian Basin. The regional fracture patterns were investigated for various lithologies in eastern Kentucky to supplement lack of structural data on outcrops in that region. Data were collected at about 150 stations and will be compiled to determine if correlations exist in jointing and fracturing among the lithologic units in different geographic locations.

Production studies initiated early in 1977 have concentrated on developing the data base for the Cottageville Gas Field which was selected as the first of three Devonian gas field exploration target areas. Data from several gas producing companies is being compiled to provide a complete record of sub-surface geology and gas production to compare with follow-up geophysical and geohydrologic investigations in the same region. Selection of a second site from prospective candidate sites is currently underway. Groundwater work was initiated in the Cottageville Field area this summer with the gathering of volumetric and water quality data. Field structure studies, delayed due to lack of available personnel, and the geophysical studies were initiated late in the fiscal year. Field work areas will be selected this fall in conjunction with USGS efforts. Equipment was obtained and modified this year for the geophysical work. Field testing will be done in the near future so exploration can begin in early summer of 1978.

**PLANS FOR THE COMING YEAR** – The structural investigations will be continued through FY 1978 with emphasis on field work in West Virginia for all tasks. Additional joint and lineament data may be gathered in Pennsylvania, Virginia, and Maryland, depending on planned USGS activi-

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ties. Regional structure contour maps will be prepared for two additional lithologic unit bases. Additional well data will be sought to extend existing maps into eastern Kentucky. Joint and fracture orientation data will be synthesized and statistical analyses performed to investigate possible joint trend correlations. Production data maps will be completed for the second gas pool study area and revisions will be made for the Cottageville Field data to reflect additional data received. For Cottageville, a final report, decline curve analyses and several cross sections illustrating the geologic relationship to production characteristics will be prepared. These data will provide the necessary information needed to evaluate results of the groundwater and geophysical field work that will be done next summer in the area. Structural mapping of specific areas to complement USGS activities will be planned in the fall and winter months and executed in the summer of FY 1978. Activities planned for FY 1978 will provide additional information necessary for development of structural data as an exploration tool for Devonian Shale Gas production. Several inter-contractor exchanges in which West Virginia University will take an active role, will also be conducted to correlate basinwide information for all contractors working on the shale project.

## **DIRECTIONAL ROCK PROPERTIES**

**WEST VIRGINIA UNIVERSITY**

**DOE - \$31,026**

**3/1/76 - Continuing**

**OBJECTIVES** – Work on this project is to measure the directional properties of Devonian Shale samples, determine their relationship to preferred direction of fracturing, and determine the feasibility of developing a directional wireline tool for in situ stress measurement and orientation of fractures. In the final analysis, an evaluation will be made on the feasibility of developing a directional property logging tool to measure the principal direction of in situ stress.

**RECENT WORK AND ACCOMPLISHMENTS** – The tasks performed under the first contract E(46-1)-8028 were completed in May 1977. The directional properties of Devonian Shale were determined through four physical property measurements: Ultrasonic velocity measurement, load test, Brazillion test, and measurement of directional elastic constants. The core specimens were taken from Well 20403 in Lincoln County, West Virginia. The core with a total of 500 ft consists of the following three sections: 2720-2836, 3402-3473, and 3710-4026 ft. General results of the laboratory core analyses show that the bedding planes are quite distinct and the most preferred direction of fractures in the bedding plane is N 60°E. Sectional composite diagrams for directional ultrasonic velocities, preferred directions of fractures, and indirect tensile strength indicate that the directional features in the horizontal plane remain almost unchanged for the three core sections. This is especially true for the results of the point load tests. Sectional composite diagrams also show that the averaged values vary with depth. The longitudinal and shear wave velocities in the first section (2700-2836) are the longest while its indirect tensile strength comes next to that of the second section. For the entire core length, the results of longitudinal wave velocity measurements, point-load and Brazillion tests carried out in the horizontal plane suggested consistently that the most preferred direction of fractures was N 60°E; next was N 90°E, and third was the N 30°E direction. This behavior can be explained by assuming that the Devonian shale contains thin lenticular microcracks lying in the horizontal plane with the long axis parallel to the N 60°E direction and the intermediate axis parallel to the N 30°W direction while the short axis is parallel to the vertical direction, i.e., the axis of the borehole. The ultrasonic velocities in the horizontal and

vertical direction showed considerable difference in that the vertical velocity was much slower. Elastic constants obtained by dynamic and static measurements indicated similar trends. The results suggest the existence of inherent planes of weakness in the horizontal plane.

**PLANS FOR THE COMING YEAR** – The ultrasonic longitudinal velocity measurements will be measured at 30 degree intervals on the core samples. The depth intervals to be measured include 3470-3700, 3000-3400, and 2836-3000 ft. Ultrasonic shear velocity measurements will be made on the same samples. Point load tests will be conducted on samples taken at each 1-ft interval over the three depth intervals. Indirect tensile strength tests (Brazilian method) will be conducted on a set of six samples taken at approximately 10-ft intervals. Directional elastic constants and Poisson's ratio will be determined at evenly spaced intervals to ascertain fracture orientation and intervals of microfracturing. Comparisons of preferred azimuth orientation from the directional properties tests with the measured induced fracture orientation from the borehole televiewer will be made to ascertain the feasibility of developing a directional wireline tool for in situ stress and preferred direction of fracture orientation.

## **EARTH FRACTURE SYSTEMS**

**WEST VIRGINIA UNIVERSITY**

**DOE - \$19,210**

**10/1/75 - 9/30/77**

**OBJECTIVES** – This study is intended to apply remote sensing techniques to the interpretation of fracture systems and structural styles in the plateau region of eastern Kentucky and West Virginia. This study will aid in the development and application of a remote sensing exploration tool for locating Devonian Shale gas, as the gas production is mainly from fracture permeability. The specific objectives are to conduct a search of pertinent literature, correlate production data with fracture and lineament patterns, analyze regional fracture patterns from remote sensing imagery, and develop a computer routine to graphically and statistically evaluate the contracted data.

**RECENT WORK AND ACCOMPLISHMENTS** – All of the specific objectives of the study, as outlined above, have been met. During this year, photolineaments were mapped from satellite and aircraft imagery, surface fractures were measured at outcrops, subsurface fractures were measured on cores, and natural gas initial open flow data were obtained from driller's logs. Fractures were found to relate to the primary directions of photolineaments; however, there was considerable scatter in these data. Wells of high initial open flows are usually found between, rather than on or photolineaments.

**FOR THE COMING YEAR** – A draft of the final report has been initiated and is expected completed on a timely basis.

## **MHF RESEARCH AND ADVANCED TECHNOLOGY PROJECT**

**SANDIA LABORATORIES**

**DOE - \$785,000**

**7/1/75 - Continuing**

**TIVES** – This work concerns the development of geophysical diagnostic techniques and t of analytical studies to determine fracture mechanics, dynamics, and geometry, and initiate

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borehole techniques for formation evaluations associated with the application of MHF technology to enhanced gas recovery. Evaluation of the geophysical diagnostic techniques will be obtained through participation in MHF experiments in western tight gas sand formations and the eastern shales. Knowledge of the fracture dynamics and geometry and formation characteristics should lead to more effective gas-stimulation technology and to more efficient reservoir production upon the commercial utilization of MHF techniques.

**RECENT WORK AND ACCOMPLISHMENTS** — Two geophysical diagnostic techniques have been evaluated for fracture characterization—a surface electrical potential technique and surface seismic recordings. The surface electrical potential technique has been successfully demonstrated as a means of providing fracture orientation and asymmetry. Six field experiments were conducted at various depths and fracture lengths to evaluate the system. Based upon the results of these tests the technique is viable for fracture lengths greater than 15 percent of the fracture depth. Periods of fracture growth during stimulation operations can also be determined. Specifications, circuitry, and software for the instrumentation system and current generator were formulated and documented. Operating companies that we have conducted fracture experiment tests with include AMOCO, CONOCO, Shell, Gas Producing Enterprises, El Paso Natural Gas, and Columbia Gas.

The design and procurement of a mobile field instrumentation laboratory was initiated. This laboratory will be used for long-term pressure testing as well as applications of new logging and formation evaluation techniques. Feasibility assessment of the surface seismic technique has been concluded with negative results. Emphasis on the seismic mapping of fracture systems has been shifted to downhole measurements. The design and fabrication of a borehole, wall-clamped, wire line, seismic system has been completed. This system will be employed in the fracture well during formation breakdown operations to record fracture signals originating close to the wellbore.

**PLANS FOR THE COMING YEAR** — The electrical potential system will continue to be used in MHF experiments to expand the data base and add knowledge of parameters that influence its performance. Emphasis on laboratory simulation experiments and analytical modeling will be increased in an attempt to derive fracture lengths from the electrical potential technique. The borehole seismic system will be evaluated in several tests to determine its applicability to measuring fracture orientation and vertical growth.

The mobile field laboratory will be deployed on several experiments. The laboratory has the capability for lowering experiments in a wellbore on single conductor, seven conductor, or slick line units. Its modular electronic equipment capabilities will include pressure measuring equipment, digital computer, and analog and multiplex data-recording capabilities.

A critical need in the western tight gas sands is a means of determining the extent of the pay sands and their properties away from the wellbore. Borehole geophysical techniques will be investigated as a means of determining lenticular lens extent, geometry, and parameters.

## SUPPORT OF MASSIVE HYDRAULIC FRACTURING RESEARCH

LAWRENCE LIVERMORE LABORATORY  
DOE - \$721,000  
2/1/76 - Continuing

**OBJECTIVES** – Theoretical and laboratory models will be developed to determine the factors which control the geometry of fractures created by the massive hydraulic fracturing technique in tight gas sands and shales. As compared with “conventional” hydraulic fracturing, MHF offers a more promising method for stimulating tight gas reservoirs in that larger amounts of fluid and proppant are pumped down the well and out into the formation to create and prop fractures at greater distances from the well. However, the geometry and interaction of MHF in the reservoir are not well understood. Since the stimulation process occurs deep within the earth, direct measurement and observation of the fracturing process in the reservoir are extremely difficult, and evaluation and interpretation must be done indirectly, usually through reservoir analysis. This analysis will yield the effects of stimulation and infer geometry where enough field data are available. A data base will be enlarged for model correlation. Existing and new geophysical logging tools will be used to evaluate pertinent reservoir parameters in the tight gas sands and to locate near-wellbore fractures in gas sands.

**RECENT WORK AND ACCOMPLISHMENTS** – Application of a two-dimensional hydraulic fracturing model has begun, along with laboratory simulation of the MHF process. Available geological and geophysical data for western tight gas sands were acquired, and low-stress mechanical characterization of Devonian shale cores has been completed. Analysis of the three-dimensional sonic logging methods and interpretation techniques for the location of natural fracturing near the wellbore in Devonian shale has been completed. A preliminary parametric analysis on the effects of fracture conductivity and azimuth on production has been completed for lenticular reservoirs.

**PLANS FOR THE COMING YEAR** – Two-dimensional hydraulic fracturing model development will continue, and development will begin on other supporting hydraulic fracturing models. Geological and geophysical data acquisition will continue. A dry hole sonic logging tool will be used in holes in Devonian shales to locate near-wellbore existing fractures, and borehole gravity information will be evaluated as a method for finding pertinent reservoir properties such as water saturation. Reservoir analysis will continue by using available field data.

## MINEBACK STIMULATION TEST PROJECT

SANDIA LABORATORIES  
DOE - \$325,000  
10/1/77 - Continuing

**OBJECTIVES** – This project provides the opportunity to contribute to the understanding of and thereby improve fracturing processes for the stimulation of natural gas production from low-permeability formations. Various stimulation techniques have been applied to these resources, such as the western tight gas sands basins and the eastern Devonian gas shales, with varying, but generally noneconomic results. Technology is based upon field production experience, laboratory testing, and empirical design models. Industry has often stated the need to perform fracture experiments in an environment that allows for direct examination of the stimulation process, thus providing for verification of or defining needed improvements in stimulation models.

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**RECENT WORK AND ACCOMPLISHMENTS** — This recently initiated project has built upon fracturing and mineback activities conducted in a tunnel complex since 1974 under a nuclear containment research program at DOE's Nevada Test Site. The feasibility of mineback testing as a means of physically characterizing stimulation processes has been demonstrated. Wells are drilled from the top of the mesa to tunnel level, thus providing an overburden of ~ 1500 ft. The created fracture system is dyed or grouted and then exposed by utilizing an Alpine mining machine.

Industry inputs have been incorporated into the planning of project activities, which included the mineback to a test conducted previously to examine sand proppant distribution during an hydraulic fracture. Observed fracture behavior deviated significantly from design. Effects of bedding and faults on fracture propagation were numerous and quantitative investigation is still underway. In another test, two hydraulic fractures were created above and below a geologic interface to specifically investigate fracture behavior at the interface between formations with different material properties. Mineback is underway. From a containment experiment sponsored by the Division of Military Applications, it was found that a radial fracture system was not produced by a contained explosive detonation. Instead, adjacent to the explosive cavity, a compacted, non-fractured region is formed, and outside this is an extensive region of reduced overburden stress where fractures can be more easily created.

**PLANS FOR THE COMING YEAR** — Evaluation of the two fractures in the interface experiment will be the major activity. Emphasis will be placed upon quantifying the fracture behavior via mineback and modeling activities. Supporting rock and fluid mechanic studies will be initiated to aid in the interpretation of observed fracture behavior, which will necessitate obtaining extensive material property and in-situ stress measurements.

Seismic data obtained during fracturing will be analyzed both as a means of mapping the fracture and as a way of understanding its propagation; mineback tests will serve as a way of "calibrating" this seismic monitoring technique via direct observation. Additional small tests from the tunnel that will investigate in-situ stress, formation breakdown behavior, and geological fracture toughness parameters as well as an evaluation of different completion techniques on fracture initiation are planned.

#### **RULISON FIELD: MHF DEMONSTRATION**

**AUSTRAL OIL COMPANY**  
DOE - \$334,000; Austral - \$599,000  
6/15/76 - 12/15/76 (Extended)

**OBJECTIVES** — This project was carried out to demonstrate the effectiveness and economics of MHF technology. Stimulation of production from large natural gas reservoirs found previously to be noncommercial because of low permeability is of great importance to the furthering of fossil energy technology. This work involved a two-stage MHF treatment and evaluation of results using an existing gas well (Federal #3-94) located in the Rulison area, Garfield County, Colorado. Upon completion of the treatments, the well produced into a pipeline to determine stimulation results.

**RECENT WORK AND ACCOMPLISHMENTS** — The experimental fracture treatment was applied in two stages treating separately the gross perforated intervals from 6198 to 6333 ft (Stage 1) and 5170 to 5630 ft (Stage 2). Approximately 542,000 gallons of gelled water and 1,140,000 lb of sand

were used. A brief cleanup flow period was allowed between stages. The fracture treatments were performed in August 1976. The treatment was designed to extend a fracture about 1400 ft from the wellbore with a propped fracture width of 0.176 inch. The production increase was expected to range from 5.5 to 6.2 times the pretreatment rate of 35 Mcf/d.

The results of the MHF treatment were poor. Production of gas was increased by a factor less than two. The average flow rate during early 1977 was 50 to 60 Mcf/d. The reason for failure of the MHF treatment in the Rulison is not completely known, although it is thought to result from both low-reservoir permeability and a restricted reservoir caused by sand lenticularity. Additional tests need to be conducted.

**PLANS FOR THE COMING YEAR** – The project is complete, and the final report has been published.

### **RIO BLANCO: MHF DEMONSTRATION**

CER CORPORATION  
DOE - \$1,975,000; CER - \$1,630,000  
6/19/74 - Continuing

**OBJECTIVES** – To test advanced hydraulic fracturing technology where it has not been possible to obtain commercial production rates, a stimulation experiment was conducted in the low-permeability, massive, gas-bearing sandstone reservoirs in the Piceance Basin in western Colorado. This test was located about 1 mile from the 1973 Rio Blanco nuclear stimulation site to permit comparison of nuclear and hydraulic fracturing techniques in this area, and thus increase knowledge of fossil fuel technology.

**RECENT WORK AND ACCOMPLISHMENTS** – The well was fractured four times, twice in the lower Mesaverde and twice in the Fort Union. A fifth zone in the Upper Mesaverde was extensively tested but abandoned as being of insufficient quality for fracturing. The self-imposed limits for fracturing required a minimum of 0.2 md-ft productive capacity or 5 microdarcies permeability in the zone. Preparatory operations were similar for each: the well was first perforated dry and the zone allowed to produce naturally for a short period. The perforated interval was then broken down with 5000 to 10,000 gallons of 2 percent KCl brine using ball sealers to assure breakdown of each perforation. This initial treatment was then cleaned up and the zone flowed for 5 to 10 days, after which the well was shut in for 2 weeks to 1 month for pressure buildup and analysis. After completion of the pre-frac analyses, the zone was given an MHF treatment followed by a cleanup and flow period of about 2 months. A packer was then set on tubing above the zone during the initial cleanup to reduce after-flow volume. After gas flow had stabilized and about 55 to 60 percent of the frac load recovered, the well was again shut in for buildup for about 2 months. The experiment has essentially been completed, lacking only a flow test of the combined zones and final analysis.

**PLANS FOR THE COMING YEAR** – Field activities on the MHF-3 well have been suspended. Further action is dependent upon making satisfactory contractual arrangements with an outside party to complete the comingling of the fractured gas zones and perform some additional testing in return for the well and subsequent gas production. A complete report will be published.

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## FORT WORTH BASIN: MHF DEMONSTRATION

DALLAS PRODUCTION, INC.  
DOE - \$150,000; Dallas Production - \$178,073  
8/1/76 - 12/31/76

**OBJECTIVES** – This field project was designed to demonstrate the effectiveness and economics of MHF technology for stimulating production from large natural gas reservoirs found to be marginal or non-commercial because of low permeability. This work included production and pressure buildup tests, a one-stage MHF treatment, and evaluation of stimulation results in an existing gas well in Wise County, Texas.

**RECENT WORK AND ACCOMPLISHMENTS** – An MHF treatment was performed on the Ferguson 1A, a marginal Bend Conglomerate gas producer, to test the feasibility of MHF that would be applicable to thousands of similar wells in the Fort Worth Basin. The proposed treatment consisted of 138,000 gallons of 65 percent foam, 100,000 lb of 100 mesh sand, 198,000 gallons of gelled water-distillate emulsion and 667,000 lb of 10-20 mesh sand. The treatment was to be pumped into five individual zones between 5957 and 6794 ft. The treatment design had 1500 ft of conductive fracture length per zone and anticipated an average productivity increase of 7.9.

The treatment commenced on September 14, 1976, and was concluded on September 16, 1976. The casing ruptured on September 14 after placing 87,000 gallons of foam, 67,000 lb of 100 mesh sand, 52,000 gallons of emulsion, and 151,000 lb of 10-20 mesh sand. Repair and cleanup operations were completed on the 16th, and an additional 51,800 gallons of foam, 38,000 lb of 100 mesh sand, 146,000 gallons of emulsion, and 250,000 lb of 10-20 mesh sand were pumped. During post-frac cleanup, only 3426 bbl of the original 7144 bbl total load were produced. Initial gas flow was 135 Mcf/d but declined to 15 Mcf/d. It appears that this technique is probably not economically feasible for this particular reservoir; however, it is possible that this conclusion is premature and not warranted on the basis of the meager data available. The mechanical problems in the well did not allow early cleanup of the frac fluids, which could contribute to the low productivity.

**PLANS FOR THE COMING YEAR** – The project is complete and the final report published.

## UINTA BASIN: MHF DEMONSTRATION

GAS PRODUCING ENTERPRISES, INC.  
DOE - \$2,827,000; GPE - \$4,932,000  
7/1/76 - 9/30/78

**OBJECTIVES** – A program is being developed to demonstrate the effectiveness and economics of MHF for stimulating production from low-permeability natural gas reservoirs. The program was carried out in the Natural Buttes Unit in the Uinta Basin, an area with large volumes of gas in place. GPE has developed MHF technology to near-commercial status and will build on the results achieved to advance the technology to commercial status.

**RECENT WORK AND ACCOMPLISHMENTS** – Natural Buttes No. 18 well was perforated in 18 4-ft zones with 1 shot/ft. The zones were fractured with 1.5 million lb of sand in 745,000 gallons of fluid. The frac job was conducted on September 22, 1976; after September 30, 1976, the well



flowed at an average of 1412 Mcf/d. The well was cleaned of sand during December and has been flowing steadily since then, but production has declined steadily to less than 1 MMcf/d.

Natural Buttes No. 19 was perforated in 19 4-ft intervals with 1 shot/ft. The frac job on September 21, 1976, consisted of 1.05 million lb of sand in 638,000 gallons of frac fluid. Production declined from 166 Mcf/d to 46 Mcf/d. Formation water production has been responsible for the poor performance of this well.

Natural Buttes No. 14 well was an existing well. On October 10, 1974, 12 sets of perforations were made at 1 perforation/ft over 4-ft intervals. The well was fractured March 15, 1977, with 1.1 million lb sand in 544,000 gallons of frac fluid. Temperature and radioactive logs were run on the fractured well and production averaged 677 Mcf/d for the month of May, 1977, representing an improvement ratio of 17.8.

Natural Buttes No. 20 was perforated in 8 zones to allow the frac job to be accomplished using the limited entry technique. The MHF was performed on June 22, 1977, using 826,000 lb of sand including 25,000 lb of glass beads in 322,000 gallons of frac fluid. Early production was at a rate of 1600 Mcf/d.

Natural Buttes No. 21 has been drilled, cored, logged and casing has been set. Natural Buttes No. 22 has been drilled to 8622 ft, and an MHF treatment has been designed and approved by DOE. Natural Buttes No. 9 has been cleaned out to 8959 PBTD, and tubing and packer run and set at 6492 ft. The MHF is being redesigned to allow treatment to be pumped down the tubing only. The design is expected to be completed early in November.

**PLANS FOR THE COMING YEAR** – Natural Buttes No. 21 will be tested extensively prior to MHF. MHF treatments will be performed on Natural Buttes No. 22 and No. 9 and under the optional program for FY 1978, CIGE No. 2 and Natural Buttes No. 23 will be fractured.

#### **RIO BLANCO: MHF DEMONSTRATION**

##### **MOBIL RESEARCH & DEVELOPMENT CORPORATION**

DOE - \$2,600,000; Mobil - \$3,967,000

7/1/76 - 12/31/78

**OBJECTIVES** – MHF demonstration experiments are being conducted in the Mesaverde Formation, Piceance Basin, Colorado, to demonstrate economic feasibility of MHF in this area. The site of the experiment is the Piceance Creek Gas Field in Rio Blanco County. A successful MHF demonstration would encourage efforts to develop the very large gas resources believed to be present in low-permeability reservoirs of the Piceance Basin and other Rocky Mountain Basins. Phase I includes drilling and completing a well with cemented casing to a subsurface depth of 10,600 ft to penetrate the Mesaverde formation. Phase II involves the design and performance of an MHF treatment and test of well productivity. In Phase III, other zones will be selected, additional MHF treatments performed, and those zones tested.

**RECENT WORK AND ACCOMPLISHMENTS** – For the first test, perforations from 10,549 to 10,680 ft were broken down by pumping 5000 gallons of 2 percent KCl water at 10 bbl/min. Measured gas flow averaged 325 Mcf/d over 3 days. This interval was fractured on June 22, 1977,

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with 580,000 lb of 20-40 mesh sand in 316,000 gallons of fluid pumped at 60 bbl/min. In addition, a pre-frac pump-in test was performed to investigate the "fracturability" of the zone. From the test results, no fracturing problems were anticipated. The fracture treatment was carried out as planned, although during the treatment a large treating pressure increase occurred that began as soon as fracturing pressure was reached and was quite large before any sand entered the perforations. This could have created a wider, shorter fracture than planned and contributed to a "screen-out" near the end of the treatment. The observed gas flow rates show an increase from 325 to 1000 Mcf/d and then leveling off to 800 Mcf/d.

A second fracture treatment was planned after extensive pre-frac testing. The treatment of the interval 9392 to 9534 ft consisted of a total of 260,000 gallons of fluid 40 to place 600,000 lb of 20-40 mesh sand. A temperature log was run immediately after the frac job with sand fill found at about 9500 ft. The job indicated that frac fluid entered both sets of perforations and that the fracture, at the well bore, did not extend much above the uppermost perforation. Gas flow was 1.2 MMcf/d on August 30, and 1.1 MMcf/d on August 31. Sand fill was cleaned from the casing and the well was killed on August 31, and an unsuccessful attempt was made to unseat the packer, set at 9360 ft. A strong flow was observed on September 1 and 2, and so additional readings were taken on September 3, 4, and 5. The readings were 3.2, 2.7 and 2.6 MMcf/d, respectively.

On September 13, the stuck packer was unseated and removed. On September 27, the flow was down to 1.1 MMcf/d because of the large amount of water remaining in the interval. The well was shut-in for pressure buildup on September 28. On October 6, the buildup showed evidence of near well bore damage (also evidenced by the reduced flow rate after cleanout), and the presence of a substantial effective fracture.

**PLANS FOR THE COMING YEAR** – A third fracture treatment is being planned. The two zones fractured will be opened to flow during the winter months, and additional zones will be tested for MHF next spring.

#### **SAND RIDGE AREA: MHF DEMONSTRATION**

**PACIFIC TRANSMISSION SUPPLY COMPANY**  
DOE - \$495,000; Pacific Transmission Supply - \$880,000  
9/1/76 - 1/31/79

**OBJECTIVES** – To evaluate the effectiveness of MHF for the stimulation of natural gas production from thick, deep, low-permeability sandstone formations, an MHF demonstration is being conducted. The Sand Ridge area is considered to have excellent gas-production potential.

**RECENT WORK AND ACCOMPLISHMENTS** – During the drilling of the well, the Mesaverde was drill-stem tested almost continuously with 18 tests being run or attempted beginning at 6560 ft. Only five misruns, resulting from packer seat failures, occurred for a 72 percent success factor. Twelve cores were cut totaling 469 ft with a recovery of 427 ft (91 percent). Four cores were oriented for stress relaxation testing by Terra Tek. Open hole mini-frac experiments were attempted for the intervals 9017 to 9032 ft and 8822 to 8830 ft with only the 9017 to 9032 ft interval being successful. An attempt to examine the resulting fracture by the use of impression packers was unsuccessful.

After perforating, acidizing, and flow testing each sand of the Mesaverde Group (Castlegate, Nelson, and Farrer Facies) that indicated gas potential while drilling or upon drill-stem testing or by analysis of all available log data, Pacific Transmission Supply Company was unable to find a zone of sufficient kh as set forth as a parameter in the contract to qualify for an MHF treatment. This kh was to be 0.5 md-ft in the Castlegate sand and 1.0 md-ft in the Nelson and Farrer facies.

**PLANS FOR THE COMING YEAR** – Since the criteria for an MHF treatment could not be fulfilled and with the verbal approval of DOE, it was concluded that the wellsite phases of the contract were met. The final report is in preparation.

#### **RIO BLANCO: MHF DEMONSTRATION**

**RIO BLANCO NATURAL GAS COMPANY**  
DOE - \$410,000; Rio Blanco Natural Gas - \$593,000  
8/1/76 - 3/31/78

**OBJECTIVES** – The Piceance Basin of western Colorado is an area containing massive gas-bearing sandstone reservoirs of low permeability. To evaluate the effectiveness of MHF for stimulating natural gas production from thick, deep sandstone reservoirs having extremely low permeability, a program of MHF tests is planned to demonstrate the potential in this promising area.

**RECENT WORK AND ACCOMPLISHMENTS** – Gas production after the first contracted MHF declined from 792 to 330 Mcf/d. After 3 months' production, the rate appeared to stabilize at a rate of 130 Mcf/d. After evaluation of the data, the decline of the flow rate within the short time suggested that the 20-40 mesh sand used may not have been a thoroughly effective proppant because of crushing or embedment.

**PLANS FOR THE COMING YEAR** – An additional MHF treatment will be conducted using essentially the identical frac materials used before, injected into the same depositional sequence of rocks, and under approximately the same reservoir conditions. The key controlled variable will be the addition of glass beads into the proppant mix.

#### **HOME FEDERAL WELL, UINTAH: MHF DEMONSTRATION**

**WESTERN OIL SHALE CORPORATION**  
DOE - \$65,000; WOSCO - \$959,089  
7/1/76 - 9/30/77

**OBJECTIVES** – This project was designed to evaluate the effectiveness of MHF for the stimulation of natural gas production from thick, deep, low-permeability sandstone reservoirs in this area of the Uinta Basin.

**RECENT WORK AND ACCOMPLISHMENTS** – The first fracture treatment was completed late in 1976. As the fracture treatment began, it was found that the injection rate at the maximum pressure of 7000 psi was only 13 bbl/min instead of the 42 bbl/min rate anticipated. Reperforation of the zone resulted in a satisfactory injection rate. The treatment used 364,400 gallons of fluid and 600,000 lb of sand. After 2 months of cleanup and attempts to flow the well, satisfactory production was not obtained, and the zone was isolated for a second stimulation attempt in sands at shallower depths.

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The second MHF treatment was conducted on December 21 using 247,500 gallons of fluid, 50,000 lb of 40-60 sand, and 450,000 lb of 20-40 mesh sand. On December 27, gas production commenced at a rate of 477 Mcf/d. The rate has essentially stabilized at approximately 155 Mcf/d in April and has remained at this average level throughout May and June.

**PLANS FOR THE COMING YEAR** – The project is complete, and the final report has been issued.

## NATURAL GAS RESOURCES IN LOW-PERMEABILITY ROCKY MOUNTAIN RESERVOIRS

U.S. GEOLOGICAL SURVEY  
DOE - \$543,170; USGS - \$100,000  
10/1/76 - Continuing

**OBJECTIVES** – This program is designed to: evaluate the total gas resource in the Rocky Mountain sedimentary basins (Northern Great Plains, Greater Green River, Uinta and Piceance Basins) by using a combination of stratigraphic, structural, geochemical, and reservoir parameters; combine the data generated with ongoing research in stimulation techniques as a guide to present and future research aimed at an accurate evaluation of the recoverable gas reserves; identify other areas having major gas resources in low-permeability reservoirs; and encode and store all generated data in an ADP system. USGS will publish all results as rapidly as possible to transfer technical information to private industry. All reports and data stored in the ADP system will be available to the public upon request.

**RECENT WORK AND ACCOMPLISHMENTS** – Technical assistance and advice on tight gas sands have been provided to Lewin & Associates, Sandia Laboratories, Booz-Allen, CER Corporation, industry personnel, and representatives of the Canadian government. Computer processing requests have been submitted to Petroleum Information and the compiling of all available geological and engineering data for all basins is underway. USGS is also doing specific technical work in the Uinta-Piceance Basin, Greater Green River Basin, and the Northern Great Plains Province.

**PLANS FOR THE COMING YEAR** – The major goals will be to determine the overall resource base of natural gas in low-permeability reservoirs and define quantities of natural gas economically recoverable for the various regions.

## TECHNICAL AND ADMINISTRATIVE SERVICES FOR NATURAL GAS PROGRAM

CER CORPORATION  
DOE - \$340,000  
4/1/76 - Continuing

**OBJECTIVES** – This activity provides technical and administrative services to DOE for the development of a multiyear program directed toward adding significant domestic sources of natural gas. The effort centers on defining a vehicle whereby technological developments could be applied to EGR from those low-permeability gas reservoirs that are currently uneconomic but which could be exploitable in the near term. The estimated resource in four western regions is 730 trillion cubic feet and economic recovery of just 3 percent of this resource would alleviate the eleven western states' supply deficiency through the year 2000 at current consumption rates.

**RECENT WORK AND ACCOMPLISHMENTS** – The Western Gas Sands Project Plan was completed, outlining an 8 year, \$150 million program to demonstrate the economic and technical feasibility of developing thick, low-permeability reservoirs found in a number of western geologic basins. Four areas were selected for primary study from the potential list: the Greater Green River, Piceance and Uintah Basins, and the Northern Great Plains Province. The principal technical activities are divided into resource assessment, laboratory R&D, and field R&D. Upon completion of the overall plan, a detailed project plan for FY 1978 was drafted that identifies specific activities and tasks that have been or are contemplated to be funded by DOE.

**PLANS FOR THE COMING YEAR** – The programmatic support provided by CER will involve technical summaries, monthly status reports, analysis of field test data, economic and environmental reviews, technical program monitoring, and the development of specific programs such as those for coring and logging interpretation. Heavy emphasis will be placed on planning in anticipation of a major field effort commencing in FY 1979. These activities will consider modification to current and new laboratory and field R&D programs, and will include project review meetings plus routine liaison and coordination with DOE Headquarters, the Energy Centers, the National Laboratories, and the USGS. Specific technical goals and requirements for future field projects will be identified as well as regional locations in anticipation of the development of RFP's for FY 1979. Where additional resource assessment or other desired information can be gained, add-on experiments will be recommended. In addition, new requirements for cores will be developed and specific areas identified.

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## PUBLICATIONS

The following articles appear in *Proceedings of the DOE Symposium on Enhanced Oil, Gas Recovery and Improved Drilling Methods, Tulsa, Okla., 30 Aug. - 1 Sept. 1977*. Tulsa: Petroleum Publishing Co., 1977:

Appledorn, C.R., and Mann, R.L. "Massive Hydraulic Fracturing Gas Stimulation Project."

Beardsley, D.E.; Wroble, J.L.; and Allen, B.W. "Pacific Transmission Supply Company, Sand Ridge Mesaverde Massive Hydraulic Fracture Project, Uinta County, Utah."

Chancellor, R.E. "Mesaverde Hydraulic Fracture Stimulation, North Piceance Basin - Progress Report."

Fitch, J.L. "Demonstration of Massive Hydraulic Fracturing, Mesaverde Formation, Piceance Basin, Colorado."

Merrill, R.G. "Determination of the Optimum Massive Hydraulic Fracturing Design for the Stimulation of the Wasatch and Mesaverde Formations." (NVO/681-1, District Category UC-92).

Short, J.A. "Review of the Natural Buttes Unit Massive Hydraulic Fracturing Project."

Spencer, C.W.; Fouch, T.D.; and Dudley, D.R. "Geological Program To Provide a Characterization of Tight, Gas-Bearing Reservoirs in the Rocky Mountain Region."

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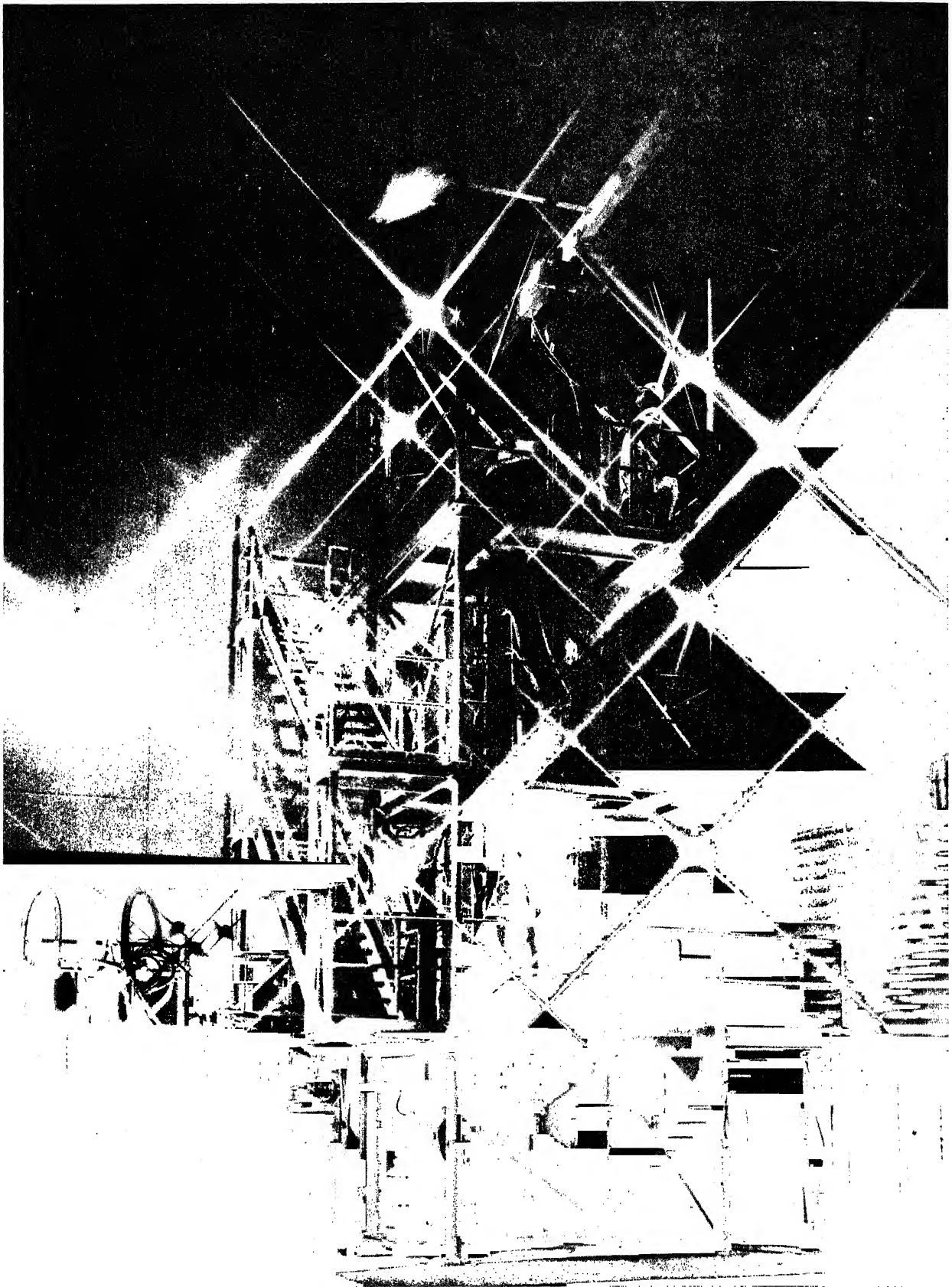
## *OIL SHALE TECHNOLOGY*

This program of research, development, and demonstration (RD&D) for oil shale technology and in situ coal gasification includes cost-shared contracts with industry and in-house research at the DOE Energy Research Centers at Morgantown, West Virginia (MERC) and Laramie, Wyoming (LERC). Projects also are conducted at Los Alamos Scientific Laboratory (LASL), Sandia Laboratories (SL), Lawrence Livermore Laboratory (LLL), Lawrence Berkeley Laboratory (LBL), and Argonne National Laboratory (ANL). Supporting research is performed at numerous universities. Rapid technology transfer is emphasized and is effected through periodic symposia, quarterly contract reports, in-house quarterly research reports, and technical presentations and publications by both DOE and contractor personnel.

The oil-shale program comprises RD&D on in situ oil-shale processes for producing both oil and gas, supporting research, and environmental studies. In-house complementary programs are conducted at the institutions listed above, with support from several universities. Four cost-shared cooperative agreements with industry have resulted in four projects on different technologies of in situ retorting of Western Green River Oil Shale and an industry contract for the in situ gasification of Michigan Antrim shale. The 1985 goal is the production of 150,000 barrels of shale oil daily by in situ retorting.

This program includes in-house underground coal-gasification projects on linked vertical wells at Hanna, Wyoming (LERC), production of medium-Btu gas through linked vertical wells and steam-oxygen injection at Hoe Creek, Wyoming (LLL), and the Eastern coal-technology project at Pricetown, West Virginia (MERC). A contract has been entered into with industry for gasification of steeply dipping beds. The 1985 goal of the program is to support production of 50,000 barrels of oil equivalent daily.

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*6-Ton Adiabatic Oil Shale Retort, Lawrence Livermore Laboratories*

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## FIELD FRACTURING AND SHALE OIL RECOVERY PROCESS DEVELOPMENT

### LARAMIE ENERGY RESEARCH CENTER

DOE - \$1,192,000

1972 - Continuing

**OBJECTIVES** — This oil shale research subprogram will develop and demonstrate technologies for the purpose of recovering an energy resource from oil shale in an economical, operationally safe, and environmentally acceptable manner. A near-term goal is to advance selected technologies to the point of demonstrating commercial application by the early 1980's. To complete this mission, research into the areas of underground shale fracturing, fracture evaluation, and oil shale retorting must be undertaken. This work is important in the development of fossil energy technology because in-situ processing of oil shale could increase the recoverable reserves by about 600 billion barrels. A technically and economically viable in situ process would reduce the water requirements of an oil shale industry and could perhaps mitigate the environmental effects normally associated with surface processing of oil shale.

**RECENT WORK AND ACCOMPLISHMENTS** — Several attempts have been made to increase the recovery of oil from the in situ experiment conducted at Site 9 near Rock Springs, Wyoming. These attempts were largely unsuccessful. The first attempt involved flushing the formation with detergents in water which resulted in production of very little oil. The second attempt involved injection of steam into the formation to mobilize shale oil and force it toward the recovery wells. This treatment appears to have promise, but in actual practice tended to plug the open spaces and required extremely high differential pressures to move liquids through the formation. Rock Springs Site 6 which was used for a fracturing and retorting experiment between 1970 and 1972 was restored. The surface casing was removed, the wells were plugged, and the site was returned to its original contours.

**PLANS FOR THE COMING YEAR** — Experimental work will be started on two additional sites during the coming year. A springing experiment will be conducted on Rock Springs Site 11, which is designed to provide void volumes for subsequent rubblizing of the oil shale. In cooperation with Sandia Laboratories a fully instrumented recovery experiment will be conducted on Rock Springs Site 12. This experiment will be similar to the recent experiment on Rock Springs Site 9, but because of improved instrumentation and changes in recovery methods, improved oil recoveries are expected.

## OIL SHALE RESEARCH

### LAWRENCE LIVERMORE LABORATORY

DOE - \$2,700,000

1973 - Continuing

**OBJECTIVES** — The oil shale program is designed to provide technology for the development of modified in situ oil shale processes, including retorting and rubblization, and to provide complementary support to joint industry/government projects, leading to the commercialization of modified or rubble in situ oil shale processing. Modified in situ oil shale has the potential to produce a liquid petroleum product at competitive prices in large volumes from a very large resource base. If developed, commercialization can be achieved by the early or middle 1980's. No other domestic source of liquid fuel has the same potential for volume production competitive with world oil prices.



**RECENT WORK AND ACCOMPLISHMENTS** – A computer model has been developed and is being used to design field experiments and for scale-up to commercial size as well as to fix priorities of laboratory experiments. Laboratory measurements of the reactions essential to the model have provided new data on char oxidation, char-carbonate decomposition, and the mechanism of oil yield degradation. The model has been used to investigate the influence of various retort parameters on oil yield, heating value of the associated gas produced, and retort rate, and has identified the most promising input air/gas mixtures. Two pilot retorts with computer controlled heat loss are in operation along with associated data acquisition systems and are providing valuable data for testing and improving the retort model. Laboratory measurements and model calculations are underway on the deformation and movement of rubble under the pressure and temperature conditions expected in the retorting environment.

**PLANS FOR THE COMING YEAR** – The working computer retort model, which calculates multiple particle sizes, will continue to be tested experimentally in our retorts and appropriate modifications made. Retort operation runs are planned with low void fraction, diverse particle size distribution, varying amounts of steam, and various inlet gas compositions. Laboratory measurements and modeling work on deformation of rubble under retorting conditions, as well as the effects on permeability and subsidence of the rubble and overburden will continue. We expect to work closely with industry in defining the key technical problems that need to be solved to make commercialization possible. In cooperation with industry, a field measurement and data acquisition effort will begin in preparation for field-scale in situ retorting.

#### **VERTICAL MODIFIED IN SITU PROJECT**

**OCCIDENTAL OIL SHALE, INC.**  
DOE - \$13,498,140; Occidental - \$5,935,860 (Phase I)  
11/1/76 - 5/1/79

**OBJECTIVES** – The work to be performed under this Agreement consists of a two-phase project. Phase I work will be performed on Occidental's Logan Wash Site, located in Garfield County, Colorado. Phase II will be conducted on the Federal C-b tract in Rio Blanco County, Colorado, in which Occidental has an interest. This work is a continuation of Occidental's research, development, and demonstration program on vertical modified in situ processes for recovering hydrocarbons and other carbonaceous values from oil shale formations. The program involves forming in situ retorts by removing a portion of the oil shale volume by mining, expanding the surrounding oil shale into the void volume by explosive fragmentation, and then retorting the fragmented oil shale in place by

More specifically, the objectives of Phase I are to (1) construct and process large-scale retorts formed by blasting to both vertical and horizontal free faces; (2) develop geologic and mine stability data for use in Phase II; (3) prepare definitive engineering designs of facilities for technical and commercial feasibility demonstrations of one of Occidental's vertical modified in situ retort designs evaluated during Phase I, utilizing data from Occidental's privately funded research and development programs on rock fragmentation, retort formation, retorting, and other areas of investigation; and (4) project commercial economics from these definitive engineering designs. Phase II will be a technical feasibility demonstration of a 2500 bbl/d demonstration plant which includes, but is not limited to, the following tasks: mining; blasting; underground construction; engineering, delivery and installation of surface facilities; shale processing, which includes pre-retorting flow tests and post retorting evaluations; and environmental studies.

**RECENT WORK AND ACCOMPLISHMENTS** — Since early 1973, Occidental Oil Shale, Inc. (OXY) has processed three small-scale modified in situ retorts (Retorts 1E, 2E, and 3E) and two large-scale retorts (Retorts 4 and 5), and is forming a third large-scale retort (Retort 6), all on OXY's Logan Wash Site in Garfield County, Colorado. Prior to the effective date of this Cooperative Agreement, November 1, 1976, OXY had mined and prepared Retort 5, in which the mined out void consisted of a vertical slot across the center of the retort and extending for the full vertical height. The overall retort dimensions were approximately 120 x 120 x 200 ft. The retort was rubbled by blasting the surrounding shale toward the vertical slot. Seismic and air shock monitoring studies were used during the rubbleing blast. Retort 5 was rubbled and processed as part of the work under this Cooperative Agreement. Prior to the ignition of Retort 5, flow and tracer tests were conducted by testing the pressure drop across the retort at various air flow rates, to determine the rate of air leakage into and out of the retort at various internal pressures, to verify the total amount of void volume as calculated from the geometry of the slot, to measure axial and radial gas dispersion, to measure gas flow distribution, and to characterize the degree and the nature of rock fragmentation in regard to particle size, particle size distribution, and the existence of channels or tight zones, etc. Ignition of Retort 5 occurred on April 18, 1977, and involved the use of shale oil as fuel for the ignition burners. Following startup, Retort 5 was processed with a mixture of air and steam. As of October 31, 1977, the total amount of oil produced in Retort 5 was 11,287 barrels. The total water produced during this same period was 130,491 barrels.

Retort 6 consists of four horizontal rooms, one above the other, separated by solid sections of oil shale. Vertical explosive holes have been drilled through the shale sections, and will be loaded with explosive just prior to rubbleing. During rubbleing, the shale in the sections between the rooms will be blasted into the mined out areas. Drilling of the explosive holes for the rubbleing of Retort 6 was started in the first week of August and was completed on October 31, 1977. The engineering and preparation of drawings for the piping, instrumentations, and miscellaneous facilities required for Retort 6 is proceeding on schedule, but the installation of the piping has not yet begun. Mine stability tests are also in progress, and initial experiments are being designed to test the strength of the spent shale remaining in the retorts. Regional environmental research plans have been completed, and environmental monitoring is continuing on both the Logan Wash tract and the C-b tract. The major marketing development effort completed under this contract has been a market analysis task subcontracted to Purvin and Gertz. The study examined in some detail transportation aspects, marketing demands, and refining considerations in the central United States. An analysis of petrochemical markets was also included as an addition to the study.

**PLANS FOR THE COMING YEAR** – Retort 6 will be rubbled, and equipment will be installed for retorting. Prior to the ignition of Retort 6, flow and tracer tests will be run to measure the pressure drop across the retort at various flow rates, and to characterize the nature of the rubbing. Retort 6 will then be ignited and processed by the introduction of a steam/air mixture. Additional Logan Wash activities will include continuing development work in the area of mine stability, and environmental monitoring and permit acquisition. The definitive engineering design for the technical feasibility demonstration facilities at the C-b tract will be completed. Also, nongovernment-funded C-b tract activities involving shaft sinking, and the construction of surface facilities will get underway as part of the necessary preparation for the technical feasibility demonstration facilities.

## TRUE IN SITU OIL SHALE PROJECT

TALLEY-FRAC CORPORATION  
DOE - \$12,436,419  
8/77 - 2/81

**OBJECTIVES** – This program is designed to determine the technical and economic feasibility of producing oil from shale with a minimum impact on the environment by true in-situ retorting. Phase I includes R&D directed toward rubbing of the oil shale and increased product recovery by use of better control instrumentation and sophisticated data acquisition systems. It is required to determine if adequate rubblization is possible by multiple explosive stimulation, if computer models can be generated to provide predictive capabilities for fracturing and retorting processes, and if a reasonable percentage of the retorted oil can be recovered. During Phase II, the demonstration phase, the objective will be to confirm the design and operation developed in Phase I.

**RECENT WORK AND ACCOMPLISHMENTS** – An Environmental Research Plan has been submitted and approved, major subcontracts let, and long-lead items ordered. The site has been surveyed, access road constructed, and mobile field office and warehouse erected. Power is being installed. A preliminary drilling plan has been submitted and approved and drilling and coring initiated. Computer models are being developed for retort design and evaluation and fracture design and evaluation. Explosive characterization is being conducted from input to computer codes. Surface and subsurface hydrology studies are being conducted and meteorology and air quality studies implemented.

**PLANS FOR THE COMING YEAR** – Injection and production wells will be drilled, the formation hydraulically broken down and explosively rubblized. Conventional recovery equipment will be installed in production wells, and flow lines connected to separators and tank battery. Flaring equipment will be put in to handle retort off gas and a use found for retort produced waters. Compressors will be installed to supply air to the retort through the injection well from which the retort will be ignited by means of a Calrod heater. Pressure and temperature measurements will be made throughout the retort, and off gases will be continuously and automatically analyzed by means of a gas chromatograph. The flame front will be monitored and controlled by means of orifice meters to maximize oil production. Throughout the program, continuing environmental studies will be conducted, including subsurface water quality analysis. The retort will be produced to exhaustion following which core samples will be taken and analyzed to determine the extent of the retorted shale. Upon completion of this effort, the site will be restored in accordance with the Environmental Research Plan.

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## BX IN SITU OIL SHALE PROJECT

### EQUITY OIL COMPANY

DOE - \$5,561,000; Equity - \$907,000

3/1/77 - 10/31/81

**OBJECTIVES** – The purpose of this work is to demonstrate the technical feasibility of using superheated steam as a heat carrying medium to retort in situ the oil shale in the Green River Formation “leached” zone, and provide a mechanism for the recovery of this oil with minimum impact on the environment. More specifically, the oil shale will be retorted by injecting superheated steam into the leached zone through an array of injection wells and recovering the steam/water/oil and gas produced from the leached zone through an array of production wells. The injection into and production from the leached zone will be accomplished in a manner which will promote a diagonal sweep of the entire leached zone. During a 2-year period, approximately  $1 \times 10^{12}$  Btu of heat will be injected into a leached zone site which is approximately 550 ft thick and covers about 1 acre. If the process can be perfected to the point of technical, economic, and environmental viability, it holds the key to the recovery of billions of barrels of oil from oil shale from a section of the Green River Formation which has been considered an unavailable resource by recovery techniques heretofore considered.

**RECENT WORK AND ACCOMPLISHMENTS** – Work under this contract was initiated on March 1, 1977. As part of a site evaluation phase, two core holes were drilled through the leached zone at Equity Oil Company's BX site near Rio Blanco, Colorado. Vertical communication tests in the form of injection pulse tests confirmed the existence of vertical communication between wells as necessary for the project. Results from the coring program and the communication tests were presented for DOE review, and approval was granted to proceed with the field project design and the laboratory experimental program phase of the contract. An environmental research plan was prepared, reviewed, and approved for the project. This plan describes and schedules the environmental monitoring which will be performed during the duration of the contract.

**PLANS FOR THE COMING YEAR** – During FY 1978, work to be accomplished on the project includes design, construction, and startup of the field project, initiation of the laboratory research program associated with the project, and initiation of the environmental monitoring associated with the project. The design/construction phase of the project should be accomplished by July 1978, with the initial steam injection or project startup beginning in July/August of 1978.

## HORIZONTAL RETORTING PROCESSES

### GEOKINETICS, INC.

DOE - \$1,022,800; Geokinetics - \$76,900 (Phase III)

11/1/76 - 12/31/77

**OBJECTIVES** – The Geokinetics research effort is being undertaken to develop economically viable processes for the extraction of shale oil from relatively shallow depths (less than 150 ft) without the

operate a number of small in situ retorts in the field. These were to range in size from 200 to 4000 tons of broken oil shale in each retort, and would provide essential design and operating data for use in scaling up to larger retorts. An environmental research program is being conducted as an integral part of the project, with the objective of evaluating the environmental effects of the processes and developing the optimum means of mitigating them.

**RECENT WORK AND ACCOMPLISHMENTS** – Ten experimental retorts were built by drilling blast holes through the overburden and the oil shale, from the surface, and detonating explosive charges in the shale bed. These experiments provided critical data for step by step improvement of the blast designs, the blast hole drilling procedures, and related effects. Five retorts or zones of broken shale, were equipped with surface process equipment, instrumentation, air access holes, oil pumps, air injection blowers, and off gas treating equipment. The rubble zones were then ignited and retorted, and the produced shale oil was recovered. Data collected from the blasting and retorting process was most satisfactory for use in scaling up the size of the experimental retorts, and almost 2000 barrels of shale oil have been produced to date. A secondary objective of the experimentation was to investigate methods of disposing of the small but steadily increasing quantities of oil that would be produced during the scale-up program. To date all crude shale oil production was sold to a local refinery, where it was successfully blended with their normal crude oil feedstock, refined into their usual products, and sold through the ordinary commercial market. The environmental research program was designed and initiated, and equipment and instrumentation were recently purchased and put into service. Base line and effects data are now being collected.

**PLANS FOR THE COMING YEAR** – Four larger retorts will be built and will represent a major scale-up from the retorts constructed during the past year. A minimum of two of these will be equipped with surface process equipment and retorted. A number of retorts already constructed will also be equipped and instrumented for retorting. In addition to the process research, a detailed research program to investigate the environmental effects of the process in actual field operation will be expanded, and methods for mitigating these effects will be developed.

## IN SITU PROCESSING OF ANTRIM OIL SHALE

DOW CHEMICAL COMPANY

DOE - \$13,927,419

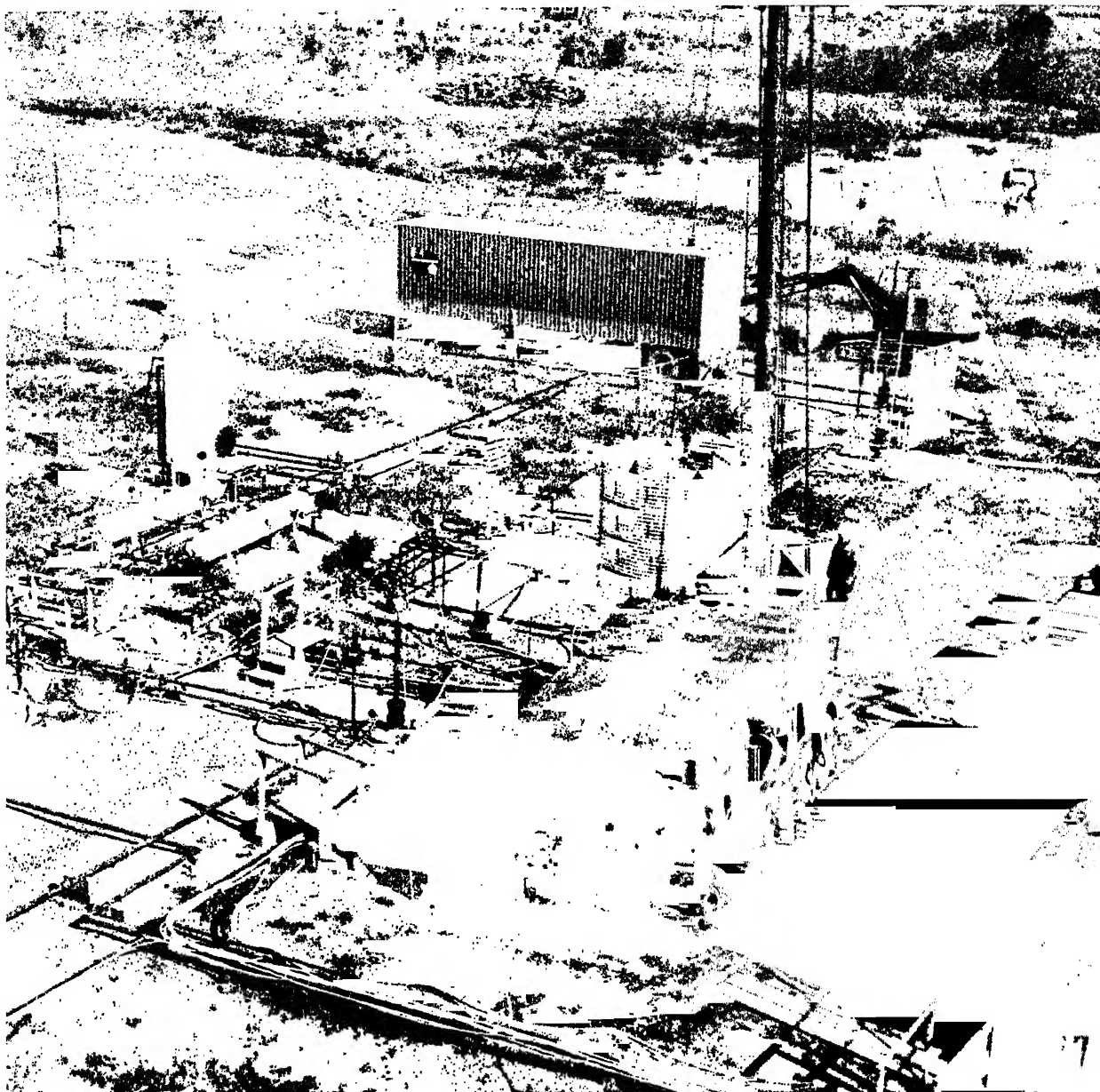
10/1/76 - 9/30/80

**OBJECTIVES** – This program will test the technical feasibility of producing energy values by in situ means from the Antrim Oil Shale, which underlies most of the lower peninsula of Michigan. Tasks to be carried out during the execution of this project include lithological, geochemical, and geophysical analysis of oil shale samples obtained throughout the Michigan Basin, and the performance of a limited number of in situ fracturing and partial combustion tests at Dow's shale test site in Michigan. The eastern Devonian oil shales, of which the Michigan Antrim is only a small part, tend to show low oil yield by standard assay techniques, in spite of the fact that they may contain an organic carbon content comparable to other shales found in the United States. These shales are also very low in carbonate content, and, therefore, the mineral matrix is not altered by normal retorting conditions. These properties, coupled with their ideal location in the industrial center of the United States, make these shales very attractive for development.



*Preliminary Core Analysis*

**RECENT WORK AND ACCOMPLISHMENTS** – On October 1, 1976, a letter contract was issued to The Dow Chemical Company to continue, expand, and accelerate a program into the technical feasibility of extracting hydrocarbons from the Michigan Antrim Oil Shale. A site in the Thumb Area of Michigan at which Dow had previously carried out a massive chemical explosive fracturing in the Antrim at a depth of 1300 feet was reactivated. Existing equipment was repaired and thoroughly tested, and new equipment was designed and purchased to dry up 10 of the 12 wells on the site. Brine removal was started in early December. A volume of 93,000 gallons of brine was removed and re-entry into the wellbores had slowed to an acceptable rate so that extensive testing of air permeability could begin. In all, about 5.5 million ft<sup>3</sup> of air were used to determine the flow patterns and communication between wells in this field. This phase of the testing had not been completed under the Dow program and was essential to better understand where materials might



*DOW Test Site*



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move under combustion conditions, and to be able to discern changes in flow patterns and permeabilities during and after combustion. Based upon the air injection data and other measurements made on the individual wells, one well was selected for attempted ignition and hydrocarbon extraction. Additional flow testing was performed in the area of this well to confirm previous observations.

Two different systems, an electric heater and a propane/air burner, are being developed to ignite the shale. Neither system has as yet proven adequate because of mechanical failures and short component life. However, in spite of these problems, evidence is that combustion was initiated on one occasion, but was not sufficient to be self-sustaining. As part of this program, three different fracturing concepts are being tested. Engineering for all three is proceeding and four new wells have been drilled, cored, and cased. Over 1600 feet of core material was collected for analysis. One well was completed to 2600 feet as an observation and seismic monitoring well, and two are to be used for hydraulic fracturing. The fourth well is to be used for chemically creating a small below-ground cavity. The first hydraulic fracturing experiment has been performed, and evaluation of results is just beginning. Core from the above wells has been submitted to Michigan Technological University, University of Michigan, and Wayne State University for measurement of lithological, geochemical, and geophysical properties. As this core material was collected in June and July, no complete data set is as yet available. Work is also progressing through the Michigan Department of Natural Resources, Michigan State University, and the University of Michigan on geologic maps and detailed stratigraphic cross sections of the Michigan Basin. In conjunction with the Dow field work, the Environmental Research Institute of Michigan is gathering extensive air, water, and soil quality data, and is assisting in fracture assessment. Sandia Laboratories is also assisting in making below-ground resistivity and temperature measurements.

***PLANS FOR THE COMING YEAR*** – Development of downhole igniter systems will continue, and several modifications to the propane/air burner will be made and tested as will other methods for ignition. As soon as ignition has been achieved in the Antrim, combustion will be carried on until sufficient data have been taken to make a reasonable technical analysis of results. Work on hydraulic fracturing will be carried through explosive stimulation and preliminary evaluation. Both of the other fracturing strategies will be in an advanced stage of development. Characterization of core material will be completed and work will proceed, based upon well cuttings, toward characterizing the Antrim of the Michigan Basin. Environmental monitoring will continue throughout field activities.

#### **ADVANCED INSTRUMENTATION AND IN SITU SUPPORT PROJECTS**

**SANDIA LABORATORIES**  
DOE - \$885,000  
12/1/75 - Continuing

***OBJECTIVES*** – This program will (1) develop advanced instrumentation sensors and measurement techniques and (2) apply these and other existing techniques to monitor, diagnose, and evaluate both the bed preparation and retorting phases of DOE-supported in situ oil shale field projects. The development of a viable in situ technology depends strongly upon instrumentation techniques to provide information on the results of bed preparation procedures being investigated, and diagnostic data on the chemical and physical mechanisms operating within the reservoir during the retorting process.



**RECENT WORK AND ACCOMPLISHMENTS** – Development has been completed on a solid-state specific oxygen sensor for use in monitoring of oxygen fugacities during retort processing. The sensor is constructed of small disks of high-conductivity, 16.9 weight-percent,  $Y_2O_3$  stabilized  $ZrO_2$  with platinum electrodes. It is made rugged by enclosing it in a ceramic jacket. Electrical leads similar to thermocouple leads permit the deployment of the sensors in the hostile environment of either surface or in situ retorts. Tests of the sensors have been conducted in laboratory retorts at Sandia and Lawrence Livermore and the 10-ton retort at LERC. Oxygen concentrations as measured by the sensors correlated well with on-line gas chromatograph analyses. An electrical resistivity technique, adapted from the standard Wenner or Schlumberger resistivity methods, has been developed for use in wellbores. The method uses a series of equally spaced point electrodes in the wellbore, which permits the measurement of apparent electrical resistivity versus depth by selecting groups of four adjacent electrodes at a time. Alternatively, apparent electrical resistivity versus radial distance from the wellbore can be obtained by increasing the spacing between selected electrodes. A preliminary reservoir flow model for describing flow through an explosively fractured oil shale bed has been developed. The model has been applied to field data taken by wellhead air flow and tracer techniques at the Rock Springs Site 6A. These data were collected in cooperation with engineers from the Laramie Energy Research Center using LERC equipment. The field data, when incorporated into the reservoir flow model, yielded a quantitative description of the effective permeability and hydraulic conductivity of the bed as rubble for retorting. Results obtained by this technique are descriptive and further suggest the improved resolution obtainable from down-hole flow logging, tracer injection and tracer detection. At the request of DOE/Fossil Energy, instrumentation and measurement techniques will be applied on DOE-supported private industry conducted in situ oil shale field projects. The objective is to provide a supplementary return of data from these field projects. Accordingly, instruments were placed in two production wellbores on the DOE/Dow Chemical Antrim Shale Project (Michigan) to monitor a retort attempt of a previously fractured site. One instrument string consists of pressure sensors and thermocouples to monitor the wellbore fluid level and the temperature of the production gases at the formation depth (~1300 ft). The other instrument string is an array of resistivity probes (described above) to monitor the advance of the reaction zone outward from the ignition well toward the monitoring well. Data are currently being collected from the instruments in both wellbores.

**PLANS FOR THE COMING YEAR** – Support on the Dow Antrim Shale Project will include instrumentation to diagnose one or more explosive fracturing experiments for retort bed preparation. Similar instrumentation support will be applied to the Talley Frac Project near Green River, Wyoming, and the Geokinetics Project near Vernal, Utah. Wellbore flow logging, tracer injection and tracer detection apparatus will be designed, constructed, and fielded. Temperature hardened systems will be developed for use in monitoring reservoir response during the retort operation. The reservoir flow model will be refined to accommodate reservoirs of arbitrary geometry. The model, field data acquisition, and data interpretation will be incorporated into a minicomputer-based data acquisition system to provide rapid interpretation of process results.

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## DIAGNOSTIC AND ROCK MECHANICS SUPPORT FOR OIL SHALE PROGRAM

SANDIA LABORATORIES  
DOE - \$625,000  
12/1/76 - Continuing

**OBJECTIVES** – Work under this project is designed to support the Laramie Energy Research Center (LERC) oil shale field program for demonstration of the technical and economic feasibility of in situ oil shale processing techniques. Support activities include site-specific characterization studies to provide input data to the design of fracturing experiments; utilization of the rock properties and appropriate explosives data in a computer simulation of the experiment to guide the experiment design; stress wave and other diagnostic measurements during the fracturing phase to determine the response of the oil shale formation to the explosive loading; post-fracture site characterization and analysis to describe the rubblization and permeability produced; and diagnostic measurements during the retorting phase.

**RECENT WORK AND ACCOMPLISHMENTS** – Analysis of data from the wellbore springing experiment at Rock Springs Site 6A has been completed. The results indicate that extensive fracturing was accomplished in the formation around the explosively charged wellbores; however, the resulting permeability was very low. Preparation has proceeded at Rock Springs Site 12 for conducting a hydraulic/chemical explosive fracture experiment to prepare the oil shale bed for retorting. The experiment includes the formation and characterization of three horizontal hydraulic fractures in the Tipton Member of the Green River Basin, and the insertion and detonation of a slurry explosive in two of the hydrofractures to rubblize and create permeability within the formation. The three hydraulic fractures were formed individually, at approximately 200 ft depth with a vertical separation of approximately 5 feet and sand-propped open. The growth of the fractures was monitored by pressure and acoustic sensors placed in outlying wellbores (up to 50 ft radius) and a sensitive tilt-meter array in shallow wellbores over the surface of the test site. Results indicate that the hydrofractures enlarged in a nominally uniform circular manner. Subsequent tests to monitor flow of water and air through the cracks indicated that the permeability was not uniform, which suggests that the sand proppant was not uniformly distributed. Elevation measurements of the hydraulic fracture intercepts at the 15 outlying wellbores indicate that the three fractures remained horizontal (parallel to the bedding planes) within  $\pm 1$  ft over the extent of the wellbore pattern. Computer simulation calculations were used extensively in the design of this experiment. The design intent calls for the insertion and simultaneous detonation of equal quantities of explosive ( $\sim 7500$  pounds each) in the bottom two fractures. In the region between the bottom two cracks, the induced shock waves reinforce and enhance the rubblization. The expanding cracks resulting from the explosive detonation provide room for expansion and shifting of the rock to provide permeability enhancement. In the region between the top sand-propped crack and the middle explosive-filled crack, the stress intensity is not as great. Fracture results on this region will be compared with the region between the two explosive cracks. Stress gages, time-of-arrival gages, and other diagnostic instrumentation have been placed in and on the formation to monitor the insertion and detonation of the explosive. Candidate slurry explosives were tested for detonability in hydraulic fractures.

**PLANS FOR THE COMING YEAR** – Insertion and detonation of the slurry explosive in the hydraulic fractures is scheduled for late in CY 1977. Characterization of the porosity and permeability achieved will proceed immediately, and the site will be prepared for a retort experi-

ment to be conducted in the summer of CY 1978. Rock mechanics design and diagnostic instrumentation support will also be provided on a rubblization experiment on Rock Springs Site 11.

## IN SITU BED PREPARATION STUDY

SANDIA LABORATORIES

DOE - \$490,000

12/1/75 - Continuing

**OBJECTIVES** – This program will determine static and dynamic mechanical response properties of oil shale and develop numerical modeling techniques to describe this response for use in the design and optimization of oil shale fracturing and retort stability. The technical feasibility of true in situ technology is dependent upon developing techniques for producing adequate fracturing and permeability within an oil shale bed. Similarly, a major problem currently facing modified in situ is achieving adequate and uniform rubblization in the retort zone.

**RECENT WORK AND ACCOMPLISHMENTS** – An extensive series of tests (over 200) have been conducted to determine the elastic moduli, deformational characteristics, and failure strengths of competent oil shale from the Green River Formation (western oil shale) as a function of kerogen content, confining (overburden) pressure, and orientation relative to the bedding planes. In order to facilitate the correlation of large amounts of experimental data from different test techniques, laboratories and experimenters, test samples have been restricted to two grades of oil shale, namely 20 and 40 gal/ton. Examination of quasistatic test results indicate that a mixture theory can be used for describing the response of oil shale of any grade, based on the properties of dolomite, the principal mineral constituent of the oil shale, and a polymer description of the kerogen. A mixture theory was previously found to be applicable for describing the high strain rate (explosive loading) response of oil shale. In particular, triaxial tests with varying confining pressures have been analyzed to yield the five elastic moduli necessary to describe transversely isotropic elastic response of oil shale. Using a well-established ASTM testing method, values of the fracture toughness (critical stress intensity factor) have been obtained and incorporated in computer codes for use in modeling field experiments. The fracture toughness results also provide the basic material data for a rate dependent model for describing the dependence of the fracture strength on loading (strain) rate. Work continues on the incorporation of all of these results into computer models. These models, in their present state of development, have been used in the simulation of the Rock Springs Site 6A and Site 12 experiments to analyze experimental results and provide experiment design guidance.

**PLANS FOR THE COMING YEAR** – Work will continue on the development of refined models for describing the mechanical response of oil shale and the incorporation of these models into the computer codes. With the strong evidence of the importance of the intermediate strain rate response of oil shale, laboratory testing will continue to obtain data in this rate regime. Deformation characteristics of jointed (prefractured) samples under confining pressure will continue to be obtained to model shock wave propagation through, and load stability of, jointed or previously fractured material.

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## MODIFIED HORIZONTAL IN SITU OIL SHALE EXPERIMENT

LARAMIE ENERGY RESEARCH CENTER

DOE - \$150,000

10/1/77 - Continuing

**OBJECTIVES** – This small-scale modified horizontal in situ experiment will design, develop, and evaluate this particular concept for oil shale processing to demonstrate its economic feasibility so that the resulting technology can be directed toward the utilization of thin (about 50 ft thick) oil shale beds under more than 100 ft of overburden. To logically achieve these objectives, mining and explosive blasting techniques, modes of retorting, and operating parameters, necessary to maximize resource utilization, must be studied in this type of experiment so that results can be scaled to a larger commercial sized operation.

**RECENT WORK AND ACCOMPLISHMENTS** – A comprehensive development plan for the entire White Mountain project was written and sent to several oil shale and petroleum companies for comments and suggestions as to direction of the research efforts. Several comprehensive replies were received and reviewed for possible technical input into the project. A coring program to determine final site location was started. A site located within Greens Canyon on White Mountain near Rock Springs, Wyoming, was tentatively chosen as the research site. To test the concept of horizontal retorting as designed for this experiment, a small horizontal trench measuring 6 ft wide, 10 ft deep and 50 ft long is currently being designed and is to be constructed at the retorting site north of Laramie. Several small-scale tests are currently being designed to define operating conditions and retorting parameters necessary for the design of the larger White Mountain project.

**PLANS FOR THE COMING YEAR** – After final site selection has been made, access roads into the area will be constructed. An existing jeep trail that passes near the site area will be widened and graveled. About 14 culverts will have to be installed over this 7 mile length of access road. Electrical power, a water well, maintenance and storage buildings, benches for adits, and propane storage facilities are to be constructed. Excavation, construction, and operation of a pilot sized retorting trench will be started in conjunction with construction work at the White Mountain site. Detailed final design of the White Mountain rubbling and retorting experiments are to be completed, and the Environmental Impact Assessment for the Rock Springs Area, including the White Mountain site, will be revised and completed.

## EXPLOSIVELY PRODUCED FRACTURE OF OIL SHALE

LOS ALAMOS SCIENTIFIC LABORATORY

DOE - \$500,000

3/1/77 - Continuing

**OBJECTIVES** – This work is designed to develop the capability to predict, control, and optimize the explosive fracturing of oil shale. This is a joint program with Sandia Laboratories, Albuquerque, with Los Alamos providing the dynamic, high strain rate rock mechanics data and quantitative explosive characterization required for computer simulation of the rubblization process. Proper resource bed preparation techniques presently represent the limiting technology for in situ retorting of oil shale. For the near future, the modified in situ technique, which provides initial void volume by standard mining operations, offers the highest promise for commercial exploitation.

**RECENT WORK AND ACCOMPLISHMENTS** – The dynamic shock wave propagation studies on oil shale of varying grades and bedding orientation are now essentially complete. A simple, two-component mechanical mixing model, the components being polymeric kerogen and the mineral matrix, appears to be quite adequate to reproduce the experimental shock-rarefaction data obtained using large diameter gas guns and explosives. Therefore, most of the material properties of oil shale are now directly related to only one parameter, the density. The fracture characteristics have been more difficult to define experimentally, however, although dynamic spall at strain rates above  $10^4/\text{sec}$  is now reasonably well determined as a function of bedding orientation and richness. Much more work is required in the intermediate strain rate regime of  $10^2$ - $10^4/\text{sec}$ , which is difficult to obtain in the laboratory but is very important for large-scale field events. A great deal of effort has been devoted to quantitative characterization of commercial explosives. Ammonium nitrate-fuel oil mixtures of varying packing densities and aluminum loadings have been studied as functions of borehole diameter, confinement, and boosting using electronic enhanced image optical framing cameras. Similar studies have been conducted on a number of commercial liquid and slurry explosives. Such studies have provided the quantitative description of the explosive detonation, expansion, and release required by the computer hydrocodes as initial and boundary conditions. They have also given considerable insight into practical criteria for the choice of existing explosives for specialized blasting applications. Hydrocode development is continuing, with most emphasis on development of suitable dynamic fracture criteria and accurate descriptions of the explosive burn. Several fracture descriptions have been incorporated into the code, including those depending on threshold resolved stress, total or shear strain, and on the more complex nucleation and growth model originally developed at SRI International. This effort is currently the most critical and active part of the program.

**PLANS FOR THE COMING YEAR** – Work on hydrocode development and the associated fracture criteria will continue at a high level. The explosive characterization effort will also continue on a programmatic basis. Most of the FY 1978 effort, however, will be devoted to planning and executing a series of three highly instrumented intermediate scale explosive fragmentation field experiments in Colorado oil shale. These experiments, which will be preceded by a series of smaller-scale cratering tests, are designed to yield information on rubblization in both horizontal and vertical modified in situ geometries, as well as insight into the special problems associated with choked blasting, or rubblization into a confined volume. The experiments do not represent optimized designs, but rather are intended to provide data to check and refine the predictive capability of our computer codes.

## **INDUSTRY PROGRAM AT ANVIL POINTS FACILITY**

**LARAMIE ENERGY RESEARCH CENTER**  
DOE - \$100,000  
1976 - Continuing

**OBJECTIVES** – This work will provide data on demonstration of the Paraho oil shale retorts at the Anvil Points Facility near Rifle, Colorado. This monitoring effort also provides data for the design of control technologies to mitigate the environmental effects associated with the surface processing of oil shale. These data will encourage further research that could eventually provide the technology for a commercial oil shale industry.

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**RECENT WORK AND ACCOMPLISHMENTS** – Two retorts were constructed at the Anvil Points Facility: the pilot plant retort is 60 ft high with a 4.5-ft outside diameter; the semiworks unit is 75 ft high and 10.5 ft in outside diameter. Until mid-1976, the Paraho Oil Shale Demonstration was privately sponsored by 17 participants at a total cost of \$9.4 million. Both retorts were operated to study process variables associated with the Paraho vertical kiln technology. Data were also collected to establish information concerning surface water and groundwater quality, stack gas emissions, and retorted shale management techniques. Baseline testing of military fuels was initiated by refining 10,000 barrels of Paraho crude shale oil under a Navy contract. Upon completion of this privately funded project, the Office of Naval Research contracted with Development Engineering, Inc., to produce and store 100,000 barrels of shale oil for further refining and product testing. The Anvil Points Facility was refurbished, and expanded operation of the semiworks retort was started on January 5, 1977. The retort has operated as long as 105 days between shutdowns to produce about 40,000 barrels of shale oil.

**PLANS FOR THE COMING YEAR** – An additional 40,000 barrels of shale oil will be produced by combined operation of the semiworks and pilot plant retorts. A computer data acquisition system will be installed to facilitate retorting research. Environmental research will be performed, and an Environmental Impact Statement for a modular retorting program will be completed.

## IN SITU OIL SHALE RETORTING: SUPPORTING RESEARCH

LARAMIE ENERGY RESEARCH CENTER  
DOE - \$545,000  
1973 - Continuing

**OBJECTIVES** – Needed fundamental and engineering data will be acquired on the kinetics of oil shale pyrolysis, shale oil transport, and retorting variables for the development of mathematical models as well as the design and development of in situ oil shale recovery processes. The development of a commercial oil shale technology requires knowledge of the processes that occur in oil shale pyrolysis. In addition, the amount of pilot plant and field-scale research needed for development of in situ shale processes can be reduced substantially by adequate mathematical models.

**RECENT WORK AND ACCOMPLISHMENTS** – The experimental program in the controlled state retort was continued. A total of ten experiments were run completing two test series: one series investigating the effect of particle size and another investigating the effects of very slow heating rates. Results from the particle size studies indicate that the smaller particle size has higher oil retention on the surface, yields a lower boiling point shale oil, and the retorted shale contains more residual carbon than larger particle size retorted at similar conditions. A new series studying shales from different geological sources has been initiated. The results obtained by retorting oil shale under a hydrogen atmosphere at elevated pressures in the pressure retort were published and a new test series investigating the effects of a carbon dioxide atmosphere was initiated. Additional tests were made using Michigan Antrim oil shales under a hydrogen atmosphere. An experimental apparatus for investigating the co-retorting of mixtures of oil shale and coal has been assembled, and a cooperative program between LERC and the University of Wyoming has been arranged to expedite the research effort. Developmental work on a global mathematical model for oil shale retorting was continued under contracts with the University of Wyoming and the Colorado School

of Mines. A laboratory apparatus has been assembled to investigate  $H_2S$  scrubbing from a gas stream using spent (retorted and burned) oil shale. Operation of this apparatus will begin as manpower becomes available. An experimental investigation of the oxidation of oil shale at sub-retorting temperatures has been completed, and the data are being analyzed for publication.

**PLANS FOR THE COMING YEAR** – Two new test series will be completed in the controlled state retort: (1) effects of shale source and (2) the effect of retorting with steam added to the retorting gas. The new project investigating the co-retorting of mixtures of oil shale and coal should be completed during the next year, and laboratory work on the  $H_2S$  scrubbing apparatus will be started. The mathematical modeling of oil shale retorting will be continued, with emphasis on development of a site specific model for a new modified horizontal in situ project.

## **SUPPORTING PROCESS DEVELOPMENT**

### **LARAMIE ENERGY RESEARCH CENTER**

DOE - \$450,000

1964 - Continuing

**OBJECTIVES** – This program aims at developing fundamental process and engineering criteria, utilizing pilot plant scale equipment, that can be extrapolated to design planning for commercial sized oil shale recovery processes. Delineation of the critical process variable is crucial to economic, as well as technical, development of any energy extraction technology. Although efforts to date have focused largely in the stimulation of an oil shale industry, research results will be applied to extraction of energy from tar sands and underground gasification of coal.

**RECENT WORK AND ACCOMPLISHMENTS** – Variables of oil shale size and assay were investigated in the 10-ton retort through completion of a series of 12 tests designed to examine potential correlation with oil recovery. The effect on retorting by upgrading the heating values of recycle gas through addition of calculated volumes of methane and further investigation of radioactive tracer techniques relatable to determination of free-path areas and particle size distribution were studied. Retorting variables of oxygen concentration and superficial gas velocity were studied during continuing retorting tests of Michigan Antrim shale. Optimization of retorting parameters is progressing. A report comparing results from an initial Antrim shale retorting test with a reference Green River shale retorting test was published. Acquisition of a limited amount of African oil shale (Moroccan) having a geological marine formation history similar to Antrim oil shale yet strikingly similar in properties to Green River oil shale provided the opportunity to conduct a comparative retorting test with other marine shales. Preliminary steam-air retorting tests of oil shales were started in the 10-ton retort. Initial tests have shown an increase in the heating value of the product gas by a factor of 4 and an oil recovery increase of about 8 percent.

**PLANS FOR THE COMING YEAR** – Steam-air retorting studies in both the 10- and 150-ton retorts will continue in an effort to determine the effect of steam addition during retorting on oil recovery, oil quality, carbon coke utilization, and stack gas beneficiation. Steam tests in the 10-ton retort will continue with saturated steam as the diluent, while a recently acquired steam boiler-superheater unit will provide superheated steam at 1300°F for the 150-ton retort. Definition of optimum retorting parameters associated with Michigan Antrim shales will continue through further experimentation. Steam effects on this eastern shale will be addressed. Compressibility studies of spent shale will be started.



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## PROCESS EVALUATION FROM PRODUCT CHARACTERISTICS

LARAMIE ENERGY RESEARCH CENTER

DOE - \$395,000

1972 - Continuing

**OBJECTIVES** – This project is designed to develop methods for defining the composition and properties of oil-shale fluid products and to apply these methods to shale oils resulting from various retorting processes to aid in shale oil recovery, processing, and utilization. This project relates directly to the goal of utilizing oil shale as a major energy source. Composition studies of the type used by this project provide necessary information to support the engineering research on the production of shale oil and/or the refining of the oil to the required products.

**RECENT WORK AND ACCOMPLISHMENTS** – Characterization studies were made on shale oils produced by various retorting processes, including true, modified, and simulated in situ and aboveground oils. Characterization of the oils involves drying, Hempel and simulated distillation analysis, component distribution by boiling point, and property determination. A data base containing the results of these analyses is maintained. A study was made to determine the variation in shale-oil quality and composition with increasing depth of burial of the oil shale. A number of biological marker compounds were identified, including isoprenoid alkanes, monocyclic terpanes, steranes, and pentacyclic triterpanes. Optical activity was shown to be a property of shale oil that indicates the degree of thermal degradation that occurs during the retorting process. Study of oils produced by various retorting methods reveals information about retorting conditions that are not observable by traditional analytical methods. Three papers presented at the Oil Shale Conversion Conference in Laramie described various phases of work on the characteristics of shale oil, compositional variations of oils with depth, and the optical activity property of shale oils. In addition, two papers were presented at the American Chemical Society meeting in Montreal and published by the Fuel Chemistry Division of that meeting.

**PLANS FOR THE COMING YEAR** – Shale oils will be analyzed, as they become available, for composition and physical property data and the results will be compiled in the shale-oil data base. A correlation will be developed between optical activity and retorting conditions required to produce a superior grade shale oil. A study will be initiated on the composition of the heavy distillates and residues of several shale oils.

## CHARACTERISTICS OF OIL SHALES

LARAMIE ENERGY RESEARCH CENTER

DOE - \$600,000

1944 - Continuing

**OBJECTIVES** – Work on this project is designed to develop and accumulate the detailed knowledge of the properties of oil shale rock and oil shale deposits essential to production of energy from oil shale. The data accumulated in this continuing project cover deposit stratigraphy and correlation; resource evaluation; mineral and organic composition; analytical methods development; and physical, chemical, and thermal properties of oil shale and how these properties vary in U.S. deposits.



**RECENT WORK AND ACCOMPLISHMENTS** – A report presenting detailed and comprehensive evaluation of the Colorado oil shale resource in the Mahogany zone and overlying oil shales, the section suitable for development by vertical modified in situ techniques, is nearing completion. Total resource in Colorado available for this type of development is measured as 419 billion barrels. Coring accomplished included drilling two coreholes in the shallow oil shales of the southern part of the Uinta Basin, three cores to extend horizontal modified in situ sites to the western Uinta Basin in Utah, and three cores to explore for a modified horizontal in situ site in Wyoming. More than 17,000 oil yield assays were added to the data library. Computer data bank storage for easy access and computation of all the Colorado corehole assay data was established and activated. Similar data banks for Utah and Wyoming have been initiated. Programs for enhancing the utility of these data banks are being developed. Thermal behavior of oil shale which has three separate mechanisms for developing fissures during heating was described. These mechanisms override thermal diffusion, permitting oil shale blocks to heat rapidly. In support of a study of mechanical properties of oil shale a 6-inch core was cut from Wyoming's Tipton member in the area of in situ field experiments. A sampling method was devised to produce the required multiple replicate samples from the 6-inch core. A modified split cylinder testing technique was devised which yields ultimate tensile strength along the bedding planes plus initial Poisson's ratio and Young's modulus values from a single test. All of these properties show high linear correlation with organic volume. Linear correspondence of organic volume with absolute rock density was demonstrated. The relationships among mineral and organic matter quantities in the oil shales of the saline depositional center were described. Circulation patterns modeled for the lake which deposited oil shale were developed, demonstrating emphatically the correspondence between known organic depositional centers and the circulation centers. Thermal analysis studies show that values for enthalpy of retorting in oil shales vary by a factor of 5 through the major oil shale stratigraphic section under test. Organic matter is not the major factor in this variation. An evaluation of high-temperature mineral reactions in in situ retorts was completed.

**PLANS FOR THE COMING YEAR** – The Colorado vertical modified in situ resource paper will be published. An evaluation of the shallow horizontal resource in Utah will be completed. High-temperature mineral reaction papers will be published. Oil shale resource and property data accumulation and use will be continued.

#### NEW PROCESS TECHNOLOGY

LARAMIE ENERGY RESEARCH CENTER  
DOE - \$300,000  
1974 - Continuing

**OBJECTIVES** – New and additional chemical structural information will be provided on the organic material present in oil shale and other carbonaceous materials. Another objective is to develop new reaction mechanisms for converting these carbonaceous materials into usable fuels in a manner that has minimum environmental impact. When the results of new conversion mechanisms appear promising, the reactions are studied at the bench-scale level until sufficient data are obtained to evaluate the merits of the reaction as to process potential.

**RECENT WORK AND ACCOMPLISHMENTS** – The carbon monoxide-water reaction with oil shale kerogen is being investigated as a possible method of converting the insoluble kerogen to a

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soluble material that can be processed into usable fuels. Previous work in a closed system and at about 6500 psig operating pressure showed that significantly high conversions can be obtained at temperatures below usual retorting temperatures. A semicontinuous flow reactor that can be operated at pressures from 200 to 1000 psig has been constructed and has been proven operational. Preliminary test runs at temperatures from 300° to 400°C and at 200 psig indicate that satisfactory conversions can be obtained at pressures much below those used previously. If these preliminary results are verified, this finding has significant implications concerning the process potential of this reaction. Seven different samples of branched-plus-cyclic alkanes have been isolated from the soluble material degraded from kerogen by reaction with CO and water at temperatures from 300° to 450°C. These fractions, which have been shown to be essentially uncontaminated with other constituents, are being studied in detail by gas chromatography/molten salt techniques. A significant number of isoprenoid and related compounds have been identified in these fractions. A gel permeation chromatography (GPC) system was developed for separating high molecular weight acids derived from kerogen oxidation or from trona brine (black waters). A mixture of nickel porphyrins was separated from the trona acids and was characterized by GPC. Occurrence of these porphyrins at relatively shallow burial depths (about 600 feet) suggests that high pressure and temperature are not essential to porphyrin diagenesis. Preliminary results of a study of the degradation of kerogen in molten salt mixtures (AlCl<sub>3</sub>-NaCl) suggest kerogen is degraded significantly at 300°C in 15 minutes. The nature of the degradation products suggests that the degradation is essentially chemical rather than thermal. Upgrading of a shale oil naphtha fraction by air oxidation at 50°C in a closed all-metal system suggests that the stability of the naphtha can be increased substantially by short-time oxidation. The mild oxidation induces maximum gum formation, which adds stability to the remaining naphtha fraction.

**PLANS FOR THE COMING YEAR** — The reaction of kerogen with CO-H<sub>2</sub>O using the semi-continuous flow reactor will continue. After sufficient tests have been made to show the feasibility of conducting the reaction at low pressures, the equipment will be used to determine if kerogen can be degraded in the system using only a solvent medium. Characterization of the CO-H<sub>2</sub>O degradation products and the oxidation degradation products will be investigated in detail. The upgrading of shale-oil naphtha by mild oxidation and the degradation of kerogen in the presence of molten salts will continue to be studied.

## CONVERSION OF SHALE OIL TO FUEL PRODUCTS

LARAMIE ENERGY RESEARCH CENTER  
DOE - \$250,000  
1973 - Continuing

**OBJECTIVES** — Petroleum refining techniques will be assessed and modified for application to shale oil, especially from in situ retorting. Commercial-scale production of synthetic crude oil to supplement our petroleum will likely make use of the huge oil shale deposits of Colorado, Utah, and Wyoming. Raw oils from these shales contain high percentages of sulfur and nitrogen compounds. To prevent detrimental effects on the environment from these compounds, and to permit the use of conventional refining processes, it is necessary to remove sulfur and nitrogen from the synthetic crude oil. Hydrogenation has been found to be an effective method for removing sulfur and nitrogen from the oil, and this study is primarily concerned with the development of improved methods of hydrogen processing as applied to shale-derived crude oil.

**RECENT WORK AND ACCOMPLISHMENTS** — Hydrogen-processing experiments were conducted with visbroken gas-combustion shale oil to supplement previous similar experiments with visbroken in situ shale oil; a commercial cobalt molybdate catalyst was used at three different space velocities with pressures of 1500 psig and below. In situ crude shale oil was dewaxed in several stages as part of a study for preparing improved hydrotreating feedstocks; by chromatographic analyses the different types of paraffinic hydrocarbons from each stage were determined. The dewaxed oil and raw in situ oil were used in a study for removing metals and other contaminants by deasphalting the oils to prepare feedstocks that will produce lower catalyst deposits during processing. A catalyst-life study in hydrotreating raw in situ crude shale oil over a commercial nickel-molybdene catalyst at 2200 psig was begun and is continuing with an on-stream period now in excess of 1200 hours.

**PLANS FOR THE COMING YEAR** — The hydrotreating catalyst-life study with raw in situ crude shale oil at 2200 psig will be completed. Downstream processing of the hydrotreated oil by hydrocracking and/or catalytic cracking to produce motor fuel will be investigated. Demetallation of shale oil by oxidation-precipitation methods and by deasphalting will be studied; upgraded shale oil will be used as feedstock for hydrogen processing experiments to determine whether lower operating pressures and/or better catalyst life will be obtained when using these "prerefined" feedstocks.

## **GASIFICATION OF OIL SHALE**

**LARAMIE ENERGY RESEARCH CENTER**  
DOE - \$500,000  
1973 - Continuing

**OBJECTIVES** — To date, the product sought to be recovered from oil shale has been shale oil, with the greatest emphasis placed on surface retorting. To assess the potential of oil shale gasification, a series of bench-scale experiments will be necessary to define process parameters and reaction kinetics, and to determine optima. Mathematical modeling will be employed in conjunction with the experiments. The determination of important process parameters will assist in characterizing the process and developing pertinent criteria for site selection and evaluation. Reaction kinetic studies will be of great value for process optimization and model development. The mathematical model will enable process description and understanding as well as being a practical tool for process control. When field-scale tests are configured, the successful model will assist in the placement of instrumentation wells and should produce the most cost effective instrumentation program. The model will also be useful in the transfer of laboratory-scale data to field-scale tests and should make real time evaluation of field tests possible. The development of surface gasifiers for oil shale should be relatively rapid due to the work already done in coal gasification facilities. In this case, oil would be produced as a liquid byproduct, not the primary product. However, mining and disposal problems should be similar to those expected for retorting.

Another objective of the small-scale laboratory experiments is to gain basic data for the characterization and treatment of effluents. This should present a unique opportunity to assess the environmental effects of the process as it is being developed, and thus shorten the time needed for commercialization. Development of this process means there will be an alternative to mining and surface retorting of oil shale. The present alternative to retorting is to leave the shale undeveloped.

**RECENT WORK AND ACCOMPLISHMENTS** — The development of the shale gasification technology is in its infancy and to date only seven laboratory experimental runs have been completed.

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Yet, these results have demonstrated the need for a more extensive research program. A new laboratory facility has been constructed and state-of-the-art laboratory equipment has been installed. The laboratory makes maximum utility of the computer system recently installed. The system software is designed to not only acquire and reduce data but to supply supervisory control to all flow controllers, adiabatic shields, and pressure controllers. This system is used for both small laboratory combustion tubes in addition to the large ½-ton combustion tube.

**PLANS FOR THE COMING YEAR** — In the near term, future work will consist of definition and refinement of the reaction kinetics and process parameters and support of the modeling effort. Preliminary work will be done in the small laboratory combustion tubes due to the short turn-around time, with refinement work being done in the large ½-ton combustion tube. Once a model has been developed, the majority of effort will be directed toward small field experimentation.

## ENVIRONMENTAL ASPECTS OF IN SITU OIL SHALE PROCESSING

### LARAMIE ENERGY RESEARCH CENTER

DOE - \$700,000

1976 - Continuing

**OBJECTIVES** — Work under this project will plan the assessment of in situ oil shale processing environmental concerns, will design and carry out subprojects to determine what oil shale fluid products or wastes might be produced and what effects they might have on the environment, and will study how these effects might be mitigated.

**RECENT WORK AND ACCOMPLISHMENTS** — Forty production and/or observation wells located at Rock Springs sites 6 and 9 and in the northern Green River Basin were routinely sampled and analyzed. All water quality data obtained during the past 3 years of this monitoring program have been computerized for use in data analysis, updating, and planning of future water monitoring programs. Statistically, significant temporal changes in water composition were observed for both Site 6 and Site 9. Meteorological, process effluent, ambient air quality, subsidence, subsurface temperature, and infrared thermographic data have also been collected at Site 9. A 1-year surface water quality baseline monitoring program for the proposed modified horizontal in situ project was completed. A large capacity refrigerated storage facility was acquired for the maintenance of water samples including 12,000 gallons obtained during the final quarter of Site 9 operation. An extensive characterization of Site 9 produced water was completed including quantitation of volatile and semi-volatile organic constituents and a 12 member inter-laboratory analysis survey for trace metals and water quality parameters. Research is in progress to assess the suitability of standard methods to the analysis of retort water constituents and to devise more accurate procedures and techniques where needed. At the present it appears that most of the accepted methods do not provide reliable data. A high performance liquid chromatographic separation system was developed to fingerprint organic materials in retort waters. Compositional analysis and speciation research afforded data on a variety of other in situ produced aqueous and gaseous effluents. Laboratory studies using Site 9 soil samples were initiated to determine the interactions and transport of organic solutes in retort water with surface and subsurface materials. This research is providing mechanistic models for interpretation of site related water monitoring data and is directed toward determining the fate and transport of constituents in released aqueous effluents. Studies to determine leaching rates, parameters influencing leachate composition, and the nature of retorted shale are continuing. Mineralogical and geochemical studies of oil shales are now in progress.

The multidisciplinary approach to the treatment of retort water has continued and a series of bench-scale treatability studies were completed. Steam stripping and weak acid ion exchange resins were demonstrated to be effective processes for the removal of alkalinity and ammonia from retort waters. Biological treatment processes, following appropriate pretreatment, showed promise for the removal of organics from retort water. Electrolytic oxidation was demonstrated as an effective roughing step for organic removal. Macroreticular resins, activated carbon, chemical treatment, and coal humic substance treatment were shown to be effective for the removal of specific trace organic and inorganic constituents. Laboratory-scale studies were initiated in most of these areas to develop design parameters for pilot plant scale-up. Based on the work completed to date, upgrading retort water for direct discharge will require the removal of oil and grease, ammonia, alkalinity, organics, salinity, and specific organics and inorganics in the order specified. A comprehensive program was initiated to determine the overall biochemical effects of aqueous effluents potentially released to the environment during in situ oil shale processing. Studies to evaluate the dose-response of aquatic biota, arid zone plants, soil microorganisms, and laboratory animals upon exposure to retort water are in progress. A fractionation scheme, which is based on a viable retort water treatment procedure, has been developed to aid in delineating toxic fractions in a manner consistent with removal of the same materials. The potential bioavailability of water-borne substances and their biodegradation are being studied as complements to the toxicity screening studies. The environmental statement of work and draft environmental research plan for Anvil Points (Paraho) have been completed. A variety of interactions with the four PON's have been developed with respect to environmental research activities. These include some collaborative research projects, site visits, and technical review, comments, and assistance in devising and implementing environmental research activities.

**PLANS FOR THE COMING YEAR** – Water quality monitoring programs will continue. Laboratory and field investigations on organic solute interactions and transport during subsurface migration of retort waters will continue, and models will be developed for interpretation of monitoring results. Studies to determine the effect of retort water on biosphere components will continue and will include a delineation of the relative toxicity of various fractions. Potential retort water treatment research will continue, and a combined economical-technical feasibility assessment will be reached. Plans will be made to pilot test a water management system. A complete effluent monitoring program will be initiated in conjunction with any new Rock Springs experiment. Verification and assessment of standard water quality analytical techniques will continue. A more intensive technical assistance effort is planned for the PON's, Anvil Points, and other opportunities that may arise. The basic program will show new areas of integration (e.g., evaluation of water treatment efficacy through biological testing) and yet will remain flexible to make optimum use of various contingencies.

## **OIL SHALE PROCESSING AND MANAGEMENT OF ENVIRONMENTAL RESIDUES**

**COLORADO SCHOOL OF MINES**

**DOE - \$94,729**

**9/1/76 - 12/31/77**

**OBJECTIVES** – This program's investigations are in three areas: first, to conduct limited leading studies to identify and quantify organic and inorganic leachates as a function of retorting conditions; second, to develop methods to treat water produced during retorting including equilibrium

studies dealing with the adsorption of organics that have been identified in the retort water, equilibrium studies of the uptake of ions on selected ion exchange resins, and a project to desorb ammonium carbonates from retort water with hot gases; and third, to investigate the sorptive properties of spent shale including adsorption studies on spent shale of hydrogen sulfide, sulfur dioxide, and other gases produced during retorting.

**RECENT WORK AND ACCOMPLISHMENTS** – Construction of a small-scale retort has been completed and spent shale is being produced. The composition and structure of the spent shale have been determined by chemical and X-ray diffraction studies. A surface area analysis of the spent shale shows that the area increases with increasing retorting temperatures up to 750°C but then decreases at higher retorting temperatures. The equipment necessary for carrying out hot gas stripping of the ammonium carbonates from retort water has been constructed. A test on a simulated retort water containing only ammonium bicarbonate showed that a 99 percent removal of these ions is feasible.

Equilibrium adsorption studies have been completed for the uptake of phenol and benzoic acid on the Rohm and Haas XAD-8 resin. The pure component and binary data are being fitted to the three-parameter adsorption model. Construction of the packed-bed adsorption apparatus has been completed. A study of the adsorption of sulfur dioxide on spent shale has been concluded. The breakthrough curves have been used to calculate adsorptive capacities and diffusion coefficients. This work shows that the uptake of sulfur dioxide on spent shale is by van der Waals forces and that the isosteric heat of adsorption is approximately equal to the heat of condensation. Intraparticle diffusion coefficients of SO<sub>2</sub> through the shale are on the order of 10<sup>-5</sup> cm<sup>2</sup>/sec and increase with increasing concentration.

**PLANS FOR THE COMING YEAR** – This contract will end December 31, 1977; however, a proposal to continue this work has been submitted.

## STEAM PYROLYSIS OF SHALE OIL

COLORADO SCHOOL OF MINES  
DOE - \$143,460; School of Mines - \$14,774  
6/76 - 8/78

**OBJECTIVES** – The oil shale deposits of the Green River Formation in Colorado, Wyoming, and Utah represent one of our Nation's largest reserves of potential fossil energy resources. A variety of processes for producing shale oil from the mineral have been developed, and better ways of changing the crude oil to useful products are now being sought. The subject investigation proposes to take advantage of the uniquely unsaturated structure of the oil for conversion to certain chemical compounds that now are in great demand by the petrochemical industry. Principal among the compounds—also known as petrochemical intermediates (PI)—are ethylene, propylene, benzene, toluene, and xylene. A method that is widely used for production of PI is that of steam pyrolysis of a hydrocarbon feed. Development of a process capable of upgrading shale oil to PI would be extremely useful by opening up a large new source of feedstocks for the petrochemical industry; therefore bench-scale steam-pyrolysis equipment will be constructed, experimental runs on certain shale oils conducted, and information developed relating operating conditions to the amount and quality of light olefin and aromatic products.

**RECENT WORK AND ACCOMPLISHMENTS** – Eighteen pyrolysis runs were completed on the two samples of crude shale oils chosen for the study—those produced in the LERC 150-ton retort and the TOSCO II retort. Desired processing conditions were maintained by control of reactor temperature, residence time, and steam-to-oil mass ratio. The 150-ton oil yielded higher weight percent feed conversions to light olefins than did the TOSCO oil, being 22 percent ethylene and 38 percent total olefins for the 150-ton compared with 20 percent ethylene and 28 percent total olefins for the TOSCO oil. In a continuation of the planned program, two more feedstocks were prepared by distillation of the crudes to produce a distillate fraction from each, amounting to the lightest two thirds of the whole crude oil. Pyrolysis runs on the distillates showed the same situation on relative yields of light hydrocarbons as noted with the crude oils; that is, greater amounts from the 150-ton distillate than from the TOSCO. The values were 29 percent ethylene and 47 percent total olefins, and 23 percent ethylene and 41 percent total olefins, respectively.

**PLANS FOR THE COMING YEAR** – Work will include construction of a new, improved reactor, preparation of additional feedstocks by hydrogenation of the distillate fractions to varying degrees of severity, steam pyrolysis runs on these feedstocks, and development of relationships among the nature of the feedstock, the processing variables, and the quality of products.

#### DEGRADATION OF ORGANIC COMPONENTS IN RETORT WATER

UNIVERSITY OF SOUTHERN CALIFORNIA  
DOE - \$65,000; University of Southern California - \$3900  
10/1/76 - 9/30/77

**OBJECTIVES** – Oil shale retort water from simulated in-situ processes is loaded with soluble materials and therefore presents serious disposal and purification problems. This investigation will provide a practical and economical method of minimizing environmental impairment and reclaiming the water from the oil shale retorting process. It will study biological and electrolytic treatments for degrading soluble organics by employing different strains of bacteria and for recovering other economically valuable materials by applying various currents or voltages.

**RECENT WORK AND ACCOMPLISHMENTS** – Studies of aerobic-activated sludge treatment showed that the reductions of chemical oxygen demand (COD) in filtered retort water ranged from 37 to 43 percent at concentrations between 100 (undiluted) and 20 percent (diluted) of retort water. Most of the reduction was achieved during the first 3 days. The aerobic treatment of retort water with mutant species (Phenobac and Polybac) indicated that after 21 days the total organic carbon (TOC) reduction for samples with sludge was 50 to 55 percent and 45 percent for those without sludge. The results of utilizing a rotating biological contactor (RBC) or biodisc for retort water treatment showed that a total 20 to 45 percent of TOC reduction was obtained from a concentration of 10 to 80 percent of retort water. The results of acclimation of bioflocculant *Desulfovibrio* to utilize the soluble organics indicated good and promising growth up to 40 percent of retort water.

Experiments on anerobic treatment indicated methane production as well as digestion of the sludge without further degradation of the aerobically treated retort water. The preliminary anerobic treatment of retort water exhibited 27 percent COD reduction. Research in electrolytic treatment provided the methodology for the purification as well as recovery of useful materials from retort



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water. Results based on a flat-plate cell treatment demonstrated that over 40 percent of the total solid residue and 80 percent of the benzene-soluble compounds were eliminated. Efficiency of this treatment was further supported by 65 percent of reduction of COD value and 92 percent of color-intensity reduction. Recovery of metals, ammonia, and carbon dioxide through the process might provide an economical basis for the synthesis of urea and other valuable products.

The basic mechanism and half-wave potentials of anodic oxidation of organics in retort water were examined by polarographic techniques. A modified extended surface electrolytic (ESE) cell was developed to treat the original and biodegraded retort water. A fluidized-bed electrode reactor was constructed to demonstrate the effective current conducting area for continuous treatment of retort water. The cost evaluation and system optimization of the electrolytic treatment of retort water was made for a 1 MMgal/d system. The results demonstrated the feasibility of the electrolytic treatment of retort water. Studies of other types of treatment including photo-oxidation, ozone-oxidation, as well as permagnate oxidation, illustrated that those processes can be used in conjunction with the other treatment processes either as pretreatment or post-treatment methods.

**PLANS FOR THE COMING YEAR** – Comparison of the major merits and limitations of each unit process, no matter biological, electrolytical, or chemical, will be made regarding the efficiency of treatment, feasibility of operation and economy. The effort to identify and to degradate the refractory components will be continued. The appropriate sequence of the treatment unit process will be tested and the best one will be recommended.

## **FOSSIL ENERGY ENVIRONMENT AND CONSERVATION CONCERNS**

**LARAMIE ENERGY RESEARCH CENTER**  
**DOE - \$190,000**  
**1976 - Continuing**

**OBJECTIVES** – Environmental and conservation concerns are vitally important to fossil energy technology development. In all cases, a concurrent environmental research plan is implemented during the process development phase. The objective of this program is environmental research to address the long-term concerns associated with in situ, modified in situ, and surface processing of fossil fuels.

**RECENT WORK AND ACCOMPLISHMENTS** – Guidelines for in situ processing research have been updated, and Environmental Research Plans and Environmental Impact Assessments for DOE/LERC projects and Industry/DOE joint funded projects have been prepared. Data input to the Environmental Impact Assessments is generated through proposals for research by university groups, from private industry, and cooperative studies designed to complement LERC in-house research. Proposals were funded to develop assessment data on revegetation potentials, toxic elements, toxicity screening, emission characterization, aqueous effects from in situ processing, transport and fate studies, and off-gas characterization and cleanup systems. Final Environmental Impact Assessments were for the in situ coal gasification project, the oil shale projects, and the tar sands project. Present work in energy conservation involves technical assistance to the citizens' conservation groups in Colorado and Wyoming. In addition, conservation programs have been supported through seminar presentations on energy conservation objectives, goals, and choices.



**PLANS FOR THE COMING YEAR** – Future plans for this program include expanded efforts in Environmental Research Plans and Environmental Impact Assessments for the third-generation process research experiments. Assessment data will be generated with Industry/DOE contracts on in situ processing for coal, oil shale, and tar sands research, as well as the modified and aboveground retorting of oil shale. Additional efforts in energy conservation will include research into the areas of energy choices and implementation of energy outreach programs.

### **BOUNDARY LAYER MODEL FOR OIL SHALE RETORTING**

**COLORADO SCHOOL OF MINES**  
DOE - \$38,000; Colorado School of Mines - \$2,047

**OBJECTIVES** – This project is designed to formulate and develop a mathematical model for the description of oil shale retorting processes. The model will differ from other mathematical models in that it will be based on a boundary layer formulation of the retorting process. Such a model offers a simple, flexible, and economical means of predicting the performance of an oil shale retorting process.

**RECENT WORK AND ACCOMPLISHMENTS** – A basic set of equations describing the physical and chemical processes that occur during the retorting of oil shale has been formulated. The “boundary layer” in the oil shale retorting problem corresponds to the high temperature reaction (combustion) zone of the retort chamber. The basic system of equations describing oil shale retorting processes is transformed into the von Mises coordinate system by the introduction of a stream variable. This transformation removes the convective term from each of the basic equations, and renders the continuity equation superfluous. Thermodynamic relations and data to describe the combustion of oil shale carbon residue have been developed. These relations and data have been incorporated into a computer program that implements the boundary layer calculation procedure. Development and testing of the computer program have continued up to the present.

**PLANS FOR THE COMING YEAR** – The thermodynamic relations and data describing the combustion of oil shale carbon residue will continue to be developed and refined. Relations permitting an improved description of oxygen- and fuel-rich combustion situations are to be introduced into the model. Development and testing of the computer program that implements the boundary layer calculation procedure are to be continued. Once the model has been fully developed and verified, it will be used as an optimization procedure to produce reaction rate constants which give closer agreement with experimental results.

### **THERMAL AND ELECTRICAL CONDUCTIVITY OF OIL SHALE**

**COLORADO STATE UNIVERSITY**  
DOE - \$64,609  
6/1/75 - 7/31/77

**OBJECTIVES** – This research aims to simultaneously measure the thermal and electrical conductivities of oil shale as a function of temperature, pressure, and kerogen content. After the measurements have been taken, a mathematical model of heat flow in oil shale will be prepared. This work will allow the estimation of the heat flowing from an in situ retort to the surrounding environment.

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Data will be used to improve existing simulations of oil shale retorting and to indicate the potential of electrical and thermal sensors to be used as retort diagnostics.

**RECENT WORK AND ACCOMPLISHMENTS** – Thermal conductivity and electrical impedance were measured on eight grades of oil shale ranging from 16 to 34 gallons per ton. Analysis of the data shows that thermal and electrical transport properties vary considerably during decomposition of kerogen, during mineral (such as carbonate) decomposition, and during loss of water from the shale. Thermal conductivity parallel to the varves is consistently higher than that perpendicular to the varves in oil shale. In samples reheated after retorting, thermal conductivity increased as a function of temperature. Dielectric absorption values are sufficiently high to permit the potential for electrical processing and prospecting. A closed form analytical model is being developed for heat flow from the retort. Solution of the equations will yield temperature distributions around the retort. General conclusions are that (1) kerogen content has a dominant effect on the electrical and thermal transport properties even at low temperatures and (2) a knowledge of mineral compositions is necessary to explain the temperature variations of transport properties.

**PLANS FOR THE COMING YEAR** – This contract came to completion during FY 1977.

## **AMMONIA AND ALKALINITY IN OIL SHALE RETORT WATERS**

UNIVERSITY OF COLORADO

DOE - \$47,930

9/1/77 - 2/28/79

**OBJECTIVES** – A thorough evaluation will be performed of the weak acid ion exchange system for removing the two major inorganic components of oil shale retort water: ammonium ions and alkalinity. This study will include an assessment of the technical feasibility, as well as a determination of system costs under optimal operating conditions.

**RECENT WORK AND ACCOMPLISHMENTS** – Work has progressed on the identification and evaluation of potentially useful weak acid ion exchange resins for removing ammonia and alkalinity from retort water. Ten resins were identified which were commercially available, and samples of eight of these have been subjected to equilibrium performance evaluations. It is planned that the remaining two resins will be evaluated in the future. In order to make an initial selection of resins for further bench- and pilot-scale studies, the relative performance of the resins was assessed in terms of capacity and resistance to fouling. The procedure used to compare the equilibrium performance involved the development of ion exchange isotherms for each resin in the virgin state. Samples of each resin were also fouled by long term exposure to retort water, and additional isotherms were developed following this exposure. In this way, comparisons could be made between the performance of the different resins, as well as a comparison of the performance of each resin before and after fouling. In brief, the results of these studies showed that there were only slight differences between resins, mainly at low concentrations. In general, there was no detectable deterioration of performance after fouling.

**PLANS FOR THE COMING YEAR** – The bench-scale study will be continued to evaluate the regeneration behavior of each of the resins. Following this, the most promising resin will be selected for pilot-scale evaluation. Bench- and pilot-scale studies will be performed to determine the optimum loading and regeneration conditions. The pilot plant system will be operated over an

extended period to characterize the product and brine quality, to determine the resin attrition rate, to identify the optimum operating conditions, and to correct any operational problems which are encountered. Following this direct treatment of oil shale retort water, the system will be evaluated for its potential in treating stripping tower effluent, and shale oil wash water.

## OXIDATION/GASIFICATION OF CARBON RESIDUE ON OIL SHALE

UNIVERSITY OF IDAHO  
DOE - \$79,480; University of Idaho - \$17,730  
2/1/77 - 1/31/79

**OBJECTIVES** – This research will determine the basic kinetics which govern the rates at which the carbon deposited on retorted oil shale can be consumed by two basic reaction schemes: oxidation and gasification. Process alternatives will be decided on the basis of this research for both above-ground and in situ oil shale process utilization of residual carbon on retorted oil shale.

**RECENT WORK AND ACCOMPLISHMENTS** – The equipment to conduct the retorting phase of the project was completed, and retorting under a variety of conditions was accomplished. This included shale assays from 15 to 50 gallons per ton, retorting rates from 0.6° to 30°F/min and purge rates from 0.03 to 0.3 scf/ft<sup>2</sup> min. Preliminary results obtained from weight loss measurements and carbon analyses indicate grade is a strong influence and that there is a slight effect of purge rate on the quantity of carbon residue deposited, but no significant effect of retorting rate. Tests were also conducted to determine whether retorted samples suffered a loss in activity when stored under atmospheric conditions. This was achieved by hydrogenating fresh and stored samples (to methane), and it was concluded that deactivation was not a problem.

**PLANS FOR THE COMING YEAR** – The characterization of the carbon residue according to its quality (i.e., activity) will be evaluated by hydrogenation and ignition temperature. The equipment to conduct the kinetic experiments will be constructed, and the first set of experiments will be aimed at devolatilization rates and oxidation rates of the residue. It is expected that normal progress will be achieved so that pertinent kinetic expressions will be obtained for both oxidation and steam gasification of the residue by the end of the year.

## LEACHING RATES FROM RETORTED OIL SHALE

TEXAS TECH UNIVERSITY  
DOE - \$136,524; Texas Tech - \$9,443  
1/20/75 - 12/31/77

**OBJECTIVES** – This research will determine the dominate mechanisms by which groundwater may leach in situ retorted oil shale. These hypotheses are demonstrated where possible by simulated in situ retorting and leaching experiments conducted in the laboratory. These investigations will assist in determining the possible environmental impact of in situ oil shale retorting, and suggest means for minimizing this impact. Samples of oil shale and groundwater are taken from specific sites so that the site specific implications of the research may be also be considered. \*

**RECENT WORK AND ACCOMPLISHMENTS** – It has been demonstrated that groundwater leaching of oil shale continues for indefinite periods of time, presumably controlled by slow

mineralogical reactions, in the resulting high pH leachates. For these tests the Colorado oil shale was retorted for 30 hours at 780°C, then leached with groundwater for up to 700 hours. The resulting pH during leaching was 12. Saturating the retorted oil shale with groundwater 55 days prior to leaching failed to significantly reduce leaching rates. It had been optimistically hoped that during the 55-day waiting period, gelatinous calcium silicates or calcium aluminum silicates would form to fill the pore space within the retorted oil shale fragments and minimize the leaching rates. X-ray diffraction studies demonstrated that analcite was destroyed and that decomposition of dolomite to calcite and periclase was the dominant carbonate reaction during retorting at 780° and 630°C. Sufficient other mineral were decomposed to cause the high pH leachate, since brucite has a negligible solubility at pH 12. Leaching experiments with Utah oil shale and groundwater are in progress. water are in progress.

**PLANS FOR THE COMING YEAR** – The Utah oil shale leaching experiments will be completed. Oil shale will be leached at near the boiling point of water to encourage formation of silicate minerals which are not formed at room temperature. To clarify mechanisms of oil shale leaching, capillary pressure curves and permeability of retorted oil shale will be determined as a function of leaching conditions. Concurrent X-ray diffraction and electron microscope observations will also be used to interpret leaching mechanisms.

## IN SITU COMBUSTION RETORTING OF OIL SHALE

UNIVERSITY OF UTAH

DOE - \$98,290

3/1/77 - 2/28/78

**OBJECTIVES** – This research aims to provide a better understanding of some of the subprocesses that occur during oil shale retorting. The type of data to be obtained will lead to construction of a more reliable engineering model of in situ retorting. Among the subprocesses to be studied are (1) the kinetics of the decomposition of kerogen and carbonate minerals using TGA equipment and, therefore, small samples to eliminate heat transfer effects; (2) the ignition characteristics of oil shale and carbonaceous residue in order to determine minimum heat flux, ignition delay time, and minimum total heat requirements; (3) the heats of decomposition of kerogen and carbonate minerals in order to estimate the total heat requirements for retorting, the rate of oil production, and the temperature profiles in the bed during retorting; and (4) the coefficient of thermal expansion of raw and spent oil shale in order to predict changes in bed permeability during retorting.

**RECENT WORK AND ACCOMPLISHMENTS** – This new project has been in operation for only part of the year. A mathematical model that incorporates the intrinsic kinetics of the decomposition of kerogen and mineral carbonates and the internal as well as the external heat transfer effects has been developed. Preliminary work indicates that, at a heating rate (1°-2°C/min) expected in a modified in situ retorting process, the model is successful for particles up to 15 cm in diameter. Oil shale ignition experiments have been carried out using an electric heating element as the ignition source on packed beds of shale. Ignition delay, minimum energy for ignition, and minimum igniter strength are being determined as functions of source temperature, airflow rate, and the size and grade of shale.

**PLANS FOR THE COMING YEAR** – Experiments will be carried out on the decomposition of kerogen and mineral carbonates. The data developed will be used to refine the mathematical model that has been derived. Work on the ignition of oil shale will continue, expanding the data base, and will include work using a natural gas flame ignition source. Work will begin on the heats of decomposition of kerogen and mineral carbonates and the thermal expansion of oil shale when equipment is set up and operating.

## **MECHANICAL PROPERTIES OF OIL SHALE**

UNIVERSITY OF WYOMING  
DOE - \$64,548; University of Wyoming - \$12,531  
2/1/77 - 8/31/78  
Principal Investigator - K.P. Chong

**OBJECTIVES** – Work on this contract will be undertaken to study the mechanical properties of Green River Formation oil shale from Wyoming's Tipton Member, comparing results with the much less fissile oil shales from the much-tested Mahogany zone at Anvil Points, Colorado. These studies facilitate all development efforts on the Tipton oil shale, now the site of several in situ projects. About half of the 1000 samples of oil shale to be tested to characterize the mechanical behavior of the Green River Formation's Tipton Member near Rock Springs, Wyoming, have been completed. The experiments include precise, representative, and duplicable sample preparation; uniaxial static compression testing; "modified" split cylinder testing; uniaxial creep and relaxation tests; dynamic tests with varying strain rates; fatigue resistance; and three-dimensional constitutive relationships. The variables investigated include organic volume, mineral content, stratigraphy, strain rates, stress levels, and time dependency.

**RECENT WORK AND ACCOMPLISHMENTS** – Presently, the testing and analyses of uniaxial compression test and "modified" split cylinder test have been completed. Uniaxial creep tests, dynamic tests with varying strain rates, and fatigue resistance tests have been finished except for the analyses and correlation. Two abstracts have been submitted for possible presentation at the 19th U.S. Symposium on Rock Mechanics, with the full manuscripts due in February 1978. Three articles have been presented at the First Annual Oil Shale Conversion Symposium, held at Laramie, Wyoming, September 1977. A full manuscript is being prepared on "modified" split cylinder test to be submitted to the American Society of Civil Engineers for review and possible publication in a technical journal. Two new testing methods have been devised: one to obtain Young's modulus, Poisson's ratio, and ultimate tensile strengths from "modified" split cylinder tests and one to derive relaxation data from creep data for small strains.

**PLANS FOR THE COMING YEAR** – Analyses and correlation of data from creep tests, dynamic tests and fatigue tests will top the agenda. Three-dimensional stress-strain characterization will probably be started in spring and carry on to summer. By August 1978 the whole project should be wrapped up.

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## MATHEMATICAL MODELS OF IN SITU OIL SHALE RETORTING

UNIVERSITY OF WYOMING  
DOE - \$99,726; University of Wyoming - \$20,752  
3/1/76 - 9/1/77

**OBJECTIVES** — This work aims to develop mathematical models to describe the recovery of oil from oil shale by in situ retorting techniques. In situ retorting techniques answer both the ecological problem of waste disposal from strip mines and the problem of extracting the ore from thick overburdens. Mathematical models of in situ retorting processes would predict oil yield from a given field site, giving planners a better idea of the utility of the field.

**RECENT WORK AND ACCOMPLISHMENTS** — A global mathematical model describing oil shale retorting originally developed by the Laramie Energy Research Center has been improved and modified to include gas channeling and oil retention. A program has been developed which will identify chemical kinetic reactions from experimental data. Another program has been developed to solve the free boundary problem for the combustion of carbon spheres.

**PLANS FOR THE COMING YEAR** — Work under this contract has been completed. The first draft of a final report on this work has been submitted to DOE.

## WATER CONSERVATION WITH IN SITU OIL SHALE DEVELOPMENT

UNIVERSITY OF CALIFORNIA  
DOE - \$24,000; DOI - \$90,000  
10/1/76 - Continuing

**OBJECTIVES** — This work will identify the effect of in situ oil shale processing on the water resources of the Upper Colorado River Basin. The work will identify both impacts and mitigating measures using a two-stage process consisting of planning and laboratory measurements. By identifying these impacts and developing control strategies and management plans before development of a commercial industry, the disruption of the oil shale region can be minimized.

**RECENT WORK AND ACCOMPLISHMENTS** — The potential impact of in situ oil shale processing on the water resources of the Upper Colorado River Basin is primarily related to two factors: water use characteristics of the technology, and the surface water and groundwater hydrology in the vicinity of the oil shale deposits. Some specific problems related to each of these two broad areas include the contamination of underground waters as a consequence of groundwater flow through abandoned in situ retorts and the disposal of effluents produced during oil shale retorting. The accomplishments of research directed at these specific problems are as follows: (1) The effect of retort operating conditions on the chemical composition of retort waters was investigated. (2) The partitioning of some 50 major and minor elements, between the spent oil shale, shale oil, retort water, and offgas was studied. (3) Continuous flow and batch laboratory studies were conducted to determine the quantity and type of organics leached from spent shale. (4) Biological treatability studies of retort water were completed. (5) A hydraulic model of the aquifer system connected by in situ retorts was developed and used to predict flow conditions through abandoned in situ retorts.

**PLANS FOR THE COMING YEAR** – The partitioning of 50 major and minor elements during in situ oil shale retorting will be studied for a range of operating conditions by using LERC's controlled state oil shale retort and by completing laboratory-scale kinetic studies. Runs using air, air/steam,  $N_2$ ,  $N_2$ /steam, and  $N_2$ /air/steam will be studied. Partitioning and speciation of Hg, Se, As, Cd, and Sb will be studied in detail using Zeeman atomic absorption spectroscopy, X-ray fluorescence, and neutron activation analysis. Scanning electron microscopy will be used in conjunction with X-ray fluorescence to determine the association of elements with particulate phases. The resulting data will be used in conjunction with geochemical and thermodynamic data to predict the partitioning of major and minor elements for a range of field retort operating conditions.

## **COAL GASIFICATION**

### **IN SITU**

#### **IN SITU COAL GASIFICATION**

LARAMIE ENERGY RESEARCH CENTER

DOE - \$2,300,000

1973 - Continuing

**OBJECTIVES** – This program is seeking to develop by 1985 a commercially viable underground gasification process for extracting energy from western coal that cannot be recovered by conventional mining techniques. Secondary objectives of economic and environmental evaluations of the technology while it is being developed are being undertaken as well as development of process control techniques.

**RECENT WORK AND ACCOMPLISHMENTS** – Three successful in situ coal gasification experiments have been completed. The Hanna II test completed in July 1976 was the most successful in situ coal gasification experiment ever conducted yielding high-quality low-Btu gas (175 Btu/scf) at high production rates (up to 12 MMscf/d), high thermal (90 percent) and process efficiencies (74 percent), and excellent resource utilization efficiency (>80 percent). Use of down-hole instrumentation to characterize the process was also successfully conducted in cooperation with Sandia Laboratories. The Hanna III test was designed, constructed, and completed during FY 1977. This test was designed to determine the environmental impacts on groundwater associated with the technology as well as offer further characterization of the in situ process. Pre-test site hydrology assessment and groundwater quality analyses were completed for the Hanna I coal seam and an overlying aquifer at the Hanna III site. The test results yielded information on the problems associated with process operation in a coal seam yielding insufficient quantities of groundwater influxing into the reaction zone. Because of this water deficiency, high wellhead temperatures (up to 1200°F) and lower gas quality (130 Btu/scf average) resulted during the 2-month test. The test did prove the advantages inherent to computer-controlled operation of the process using control loops and valves to maintain stable air injection rates and reservoir pressures. While Hanna III was being conducted, the design of the Hanna IV test was completed and construction was begun. The Hanna IV well pattern consists of three wells in a line with spacings of 100 and 150 feet in order to demonstrate successful operation at greater well spacings which significantly affects the economics of the process. Hanna IV represents a significant increase in size over previous tests.

**PLANS FOR THE COMING YEAR** – The primary task will involve the conduct of Hanna IV. The estimated duration is 6 months with gasification of 28,000 tons of coal (approximately twice the

amount during all previous Hanna tests) and production rates up to 27 MMscf/d with injection rates as high as 19 MMscf/d. In addition, the product gas will be monitored for particulate matter, trace metals, and trace components ( $\text{CS}_2$ , COS,  $\text{NH}_3$ , and HCN). Subsidence measurements will also be made to determine effects on the process (e.g., gas leakage due to subsidence) as well as associated environmental impacts. Collection and analyses of byproduct hydrocarbon liquids will also continue to determine the value of these liquids, which account for 5 percent of the total energy output from the process. Modeling of the process, which has been very successful to date in predicting process results, will continue with laboratory simulation of the process being undertaken. Samples of produced water will be characterized, and a large sample of the produced water (>5,000 gallons) will be collected for use in biochemical effects and related health studies to further elucidate any associated environmental impacts and occupational health hazards. Post-test water sampling and analyses at the Hanna III site will continue. These data will be used to determine changes in groundwater quality within the coal seam aquifer and an overlying aquifer, to determine the dispersion through the groundwater regime of added constituents, and to determine the sorption capacity of unaffected coal as a measure of retardation of contaminant dispersion. The results of Hanna IV will be used to design Hanna V, a nine-well pattern approaching the pilot plant development stage. Based on Hanna IV the well spacings and operating parameters will be adjusted to yield optimal results representative of commercial operation.

## PROCESSES FOR IN SITU COAL GASIFICATION

LAWRENCE LIVERMORE LABORATORY

DOE - \$2,700,000\*

7/1/75 - Continuing

**OBJECTIVES** — A commercial in situ coal gasification process will be developed which produces medium-Btu gas suitable as a chemical feedstock or for upgrading to pipeline quality or production of liquids. Linkage methods to enhance the permeability of the coal which are under investigation include chemical explosive fracturing, shaped charges, directional drilling, and reverse combustion. The resultant permeable coal would be gasified with mixtures of oxygen and steam or carbon dioxide, and the gases would be cleaned and upgraded at the surface. The process would be applicable to deep, thick western coal. In situ coal gasification offers three major potential advantages as a source of synthetic fuel: lower costs, utilization of resources that are uneconomical for strip or deep mining, and environmental advantages. These include the avoidance of surface disruption and contamination characteristic of surface mining, and elimination of the need to subject personnel to underground mining hazards. Surface wastes are not expected to be a problem; contaminated water would be returned underground. If the project is successful, commercial quantities of synthetic gas or clean syngas for chemicals will be available in the mid- or late-1980's.

**RECENT WORK AND ACCOMPLISHMENTS** — Previously, the project has involved development work on the packed bed process, using explosive fracturing. A field gasification experiment (Hoe Creek 1 in Wyoming's Powder River Basin) has been conducted where coal was ignited and gasification proceeded for 11 days. An undesired distribution of permeabilities caused a limited resource recovery, and work is being continued in the laboratory to develop a better model for prediction of permeabilities produced by explosive fracturing. Goals have been changed to the use of reverse combustion and shaped charges as the highest priority linking methods. Field

\*Funding is for FY 1977.



preparations have been completed for Hoe Creek 2, a reverse burn field test with wells spaced 60 ft apart near the site at which the first experiment was conducted. Shaped charge tests at a coal outcrop in Wyoming have indicated that a 12-inch charge could penetrate 25 ft of coal, and a tandem shaped charge has been designed which might provide 50-ft penetration.

**PLANS FOR THE COMING YEAR** – Gasification of Hoe Creek 2 will be carried out during October-November 1977; a three-day steam-oxygen burn will also be conducted during this period to gain valuable operating experience in a coal outcrop and downhole at Hoe Creek. Hoe Creek 3, a test using only steam and oxygen and an increased amount of gasified coal, will be designed and fielded. Limited operation of the adiabatic laboratory reactor to gasify coal and the development of a process model will continue. Subsidence modeling will be incorporated into both the process and environmental models. Environmental studies, economic models, and critical analysis of extensive Soviet underground data will continue.

## UNDERGROUND COAL GASIFICATION

### MORGANTOWN ENERGY RESEARCH CENTER

DOE - \$1,100,000

1/73 - Continuing

**OBJECTIVES** – The MERC underground coal gasification project for eastern bituminous coals will develop a viable technique for the production of low-Btu gas using new techniques and advanced technology. Coal resources that might otherwise be difficult to recover because of partings, strata inclination, or low quality could lend themselves to in situ conversion. The demonstration will include tests of the Linked Vertical Well (LVW) concept and of the Longwall Co-Flow Stream (CFS) concept, as well as the latest technology in tracking and control of burn patterns.

**RECENT WORK AND ACCOMPLISHMENTS** – Three major activities, well drilling, surface hardware design, and environmental planning, have been initiated at the field site at Pricetown, West Virginia. Progress has been made in each case. Preliminary well drilling for the CFS test now includes a horizontal well in the coal, achieved by directional drilling, and two vertical observation wells. Drilling completed for the LVW test includes three injection/production wells, four monitoring wells, and one hydrology well. Wells that remain to be drilled for the upcoming LVW test include a core well and additional hydrology wells. The surface piping design for the LVW test will be completed in the second quarter of FY 1978. The formulation of a reasonable environmental plan has been coordinated by a team of TRW/West Virginia University personnel and monitored by MERC. This planning is in the final stages, and preliminary tests have been initiated. A final report on the laboratory simulation burns has been received, and presentation of these results has been made to DOE management. A reverse combustion linking model, which appears to predict much of the laboratory data, has been completed. Several meaningful tests and laboratory improvements have been identified, and a decision has been made to include additional laboratory testing in the program plan.

**PLANS FOR THE COMING YEAR** – Current plans include completion of (1) a field drilling program, (2) surface piping system design and construction, and (3) a subsurface instrument package along with a data acquisition system. The laboratory simulation data report, which indicates that meaningful tests are desirable, will be reviewed with the intent to obtain supplemental

funds for this phase of the program. The final report of the first phase of the gasification process model has been completed, and a continuation of the modeling has been proposed.

## IN SITU COAL GASIFICATION

SANDIA LABORATORIES

DOE - \$1,340,000

12/74 - Continuing

**OBJECTIVES** — The objectives of this work are to evaluate instrumentation techniques as they apply to in situ coal gasification; to obtain diagnostic information on the chemical and physical mechanisms associated with this process; to develop and evaluate remote monitoring techniques and establish their correlations with the process; and to develop a process control system that utilizes remote monitoring techniques to optimize the recovery of energy by in situ coal gasification. This program addresses the fact that successful development of in situ technology will require a thorough understanding of the chemical and physical mechanisms of the in situ process and its relation to the resource and geology, along with a positive process control to obtain a product of consistent quality and quantity with efficient resource utilization. Results from this program should have direct application to the development of in situ combustion recovery techniques for other fossil energy resources such as oil shale, tar sand, and petroleum.

**RECENT WORK AND ACCOMPLISHMENTS** — The program has been associated with the Laramie Energy Research Center's in situ coal gasification program and the field experiments being conducted near Hanna, Wyoming. In situ diagnostic and remote monitoring techniques are being developed. The primary diagnostic information is obtained from extensive thermocouple arrays; gas sampling canisters, tilt meters, and displacement gauges are also being used. Surface or remote instrumentation techniques under development include electrical potential and resistivity, acoustic, and seismic techniques. The instrumentation effort (15 instrumentation wells, extensive surface probe arrays, and over 600 recording channels) fielded on the Hanna II experiment in 1975-1976 made that test the most thoroughly instrumented in situ coal gasification test yet conducted. Subsequent thermal data analyses have provided an excellent characterization of the movement and structure of the reaction front, and these results have been incorporated into improved process models. An assessment of all instrumentation techniques fielded on Hanna II has been completed; the techniques, their hardware and fielding, results, and future development directions have been presented in a comprehensive report. In general, positive results were obtained. Based on this assessment, a field instrumentation effort was defined for the Hanna IV experiment and was fielded April-August 1977. Thirty diagnostic wells containing special branched thermocouples and gas sampling canisters were fielded within the 120 x 300 ft experimental area. An additional well to the top of the coal seam will examine the upward progression of subsidence via a modified reflectometry technique. A 300 probe surface array will measure electric potential changes produced by the conductive reaction region for two different current electrode configurations. Geophones in six of the wells will be used to locate the source of signals produced by cracks and sags in the overburden. State-of-the-art, high-resolution seismic reflection surveys will also be run before and after gasification. A field data system built around a minicomputer has been designed, built, and fielded and will be used for all techniques to provide on site, real time data acquisition, reduction, storage, and display.

**PLANS FOR THE COMING YEAR** – The Hanna IV test is scheduled to begin in November 1977 and has an expected 6 to 8 month duration; all techniques will be monitored during the test. Thermal data acquisition, validation and analysis, and inputs from the other techniques will be used to provide additional data to LERC for the day-to-day conduct of the experiment. A reassessment of all techniques will be made from results of this test. A diagnostic well specification, based upon Hanna IV design and experience, will be finalized for transfer to industry. Preliminary design and partial fielding for the future Hanna V test will occur. Longer term activities will focus upon development of other advanced instrumentation techniques and development of a total instrumentation capability for process monitoring and control.

## **IN SITU GASIFICATION - REACTION KINETICS**

**ARGONNE NATIONAL LABORATORY**

**DOE - \$150,000**

**1977 - Continuing**

**OBJECTIVES** – This program will provide kinetic data for the chemical reactions that occur during underground coal gasification (UCG) and determine the important processing parameters for the control of these reactions. These data will be provided in a form suitable for use in mathematical models of the in situ gasification processes currently being developed at the other DOE laboratories. A thorough understanding of the chemical reactions is necessary for the use of mathematic models to predict product quality and motion of the reaction fronts underground. Tailoring of the composition of the products from this process will be necessary for maximum utilization for a variety of end uses, which could include use of the gas as boiler fuel for electric power or process heat generation, combined-cycle power generation, or production of petrochemicals.

**RECENT WORK AND ACCOMPLISHMENTS** – Studies have been completed pertaining to the reaction of steam with Wyodak and Hanna western subbituminous coals. The dependence of the reaction rates on temperature, partial pressure of steam, and partial pressure of hydrogen has been determined. Although the Wyodak char is approximately twice as reactive as the Hanna char under similar reaction conditions at differential conversion rates, the reaction of steam with the Wyodak char is more severely inhibited by hydrogen than with the Hanna char. As a result, under expected operating conditions in an underground gasification zone, the rate of reaction of steam with the Hanna char will generally be equal to, or greater than, that for the Wyodak char. We have determined that the mineral matter present in these western coals is indeed an excellent catalyst for the water gas shift reaction, and for contact times as short as a few seconds, this reaction approaches thermodynamic equilibrium at approximately 550°C. We have also studied the catalysis

**PLANS FOR THE COMING YEAR** – We have recently begun studies of the reaction of steam with chars prepared from Pittsburgh seam bituminous coal. The physical and chemical properties of this char are quite different from those of the western coals, and as a result the reaction characteristics are also very different. This work will be completed, and the reaction of all three chars with carbon dioxide will be investigated. In addition, reaction characteristics of lignite char will be studied.

## COMBINED $\text{CO}_2\text{-O}_2$ PYROLYSIS GASIFICATION FOR SOUTHWESTERN COALS

LOS ALAMOS SCIENTIFIC LABORATORY

DOE - \$80,000

4/1/77 - Continuing

**OBJECTIVES** – This work seeks to demonstrate the technical, economic, and societal parameters of a two-stage underground  $\text{CO}_2\text{-O}_2$  blown coal utilization scheme specifically designed for those conditions prevailing in the arid regions of the southwestern United States.

**RECENT WORK AND ACCOMPLISHMENTS** – Although currently the southwestern United States is a major producer of petroleum and natural gas, production of these resources is in decline. Vast quantities of subbituminous coals are in this region; large surface coal fired facilities now use this fuel. A rapid expansion in coal utilization may be limited by a series of environmental problems unique to the region, especially water availability. Moreover, much of the identified coal is too deep for surface extraction. Underground coal gasification is markedly influenced by underground water intrusions. Because of limited intrusion, the dry seams in the Southwest would yield a significant fraction of energy during underground gasification as sensible heat in the producer gas. This heat, in the LASL scheme, is transferred to another, adjacent underground coal section causing thermally induced shrinkage and pyrolysis. The pyrolysis, driven by the normally wasted sensible heat leads to the removal of a separate hydrogen-enriched stream of low molecular weight hydrocarbon gases and liquids and leaves underground a hot char for subsequent gasification. This overall process requires minimal water consumption and permits the utilization of deeply lying coals within existing air-shed quality restrictions. The central underground problem is to control a complex heat and mass transfer process within a coal seam. Studies underway explore the underground transfer mechanisms. Laboratory measurements have been made using both ceramic materials of known mass transfer resistance and sections removed from southwestern subbituminous coal seams. These materials have been instrumented and then heated, using forced convective transfer, to determine the effects of moisture removal on the mass transfer processes. Heat transfer, upon drying, changes from a conductive to a convective regime. These data, along with computer modeling of the combined heat and mass transfer problem, suggest that underground thermal conversion of coal is feasible if water intrusions into the process region are not significant.

**PLANS FOR THE COMING YEAR** – Work will continue on the mechanics of heat transfer using representative coal blocks. The drying stages are capable of accurate description. Pyrolysis reactions will be explored next to identify rates of production and rates of mass transfer of the produced gases. These studies will generate input which will permit continued refinement of mathematical models that can address the underground situations. Work will continue on planning for field tests that will thermally treat large confined sections of an underground coal seam. Such controlled field experiments are necessary to adequately test the modeling of these in situ heat and mass transfer processes.

## PYROLYSIS OF LARGE COAL BLOCKS

OAK RIDGE NATIONAL LABORATORY

DOE - \$150,000

1974 - Continuing

**OBJECTIVES** – The purpose of this program is to improve the performance of in situ gasification field tests via obtaining improved data for modeling. Chemical engineering aspects of the pyrolysis step of underground coal gasification are being evaluated, and pyrolysis product yields of coals from the field test are being examined.

**RECENT WORK AND ACCOMPLISHMENTS** – Simulation of the in situ pyrolysis step has consisted of experiments with large, instrumented coal blocks. These experiments have used both subbituminous coals, supporting field tests at Laramie Energy Research Center, and bituminous coals, supporting Morgantown Energy Research Center. Cut into a 6-in. right circular cylinder, the coal is drilled with fine holes for internal thermocouple placement. Maximum reactor temperatures 500° to 1000°C have been studied, using heating rates of 0.3, 3, and 14 C°/min. From these experiments, data have been produced which correlate tar and gas production rate and composition with heating rate and maximum temperature. The first phase of experimentation focused on subbituminous coal. High natural water content (30 percent) was shown to markedly affect pyrolysis gas production, gas flow rate, and potential resource recovery. From measurements of thermal profiles, vaporization of water was shown to proceed slowly despite high surface heating rates, becoming the rate-limiting mechanism for internal heat transfer. Also, as this steam diffused out from the wet core to the hot surface, block self-gasification occurred and H<sub>2</sub> and CO were produced in amounts substantially exceeding those from dried blocks or powder samples. In all subbituminous coals, pyrolysis strongly increased solid reactivity, producing pyrophoric chars in each experiment. Bituminous coal block pyrolysis has been the focus of the second phase effort. Structural swelling and increased production of light and heavy hydrocarbons are affected by heating rate and maximum temperature in block pyrolysis. In contrast to subbituminous coals, bituminous coals contain much less water, decreasing self-gasification effects; however, hydrocarbon vapors must, like the steam, diffuse through the hot block which offers significant mass transfer resistance. Because of this, the vapors are cracked to H<sub>2</sub> and lighter hydrocarbons. Swelling of the blocks was observed to be strongly dependent on heating rate, decreasing with increased heating rate.

**PLANS FOR THE COMING YEAR** – To support in situ gasification field tests and modeling, the data base describing in situ pyrolysis effects will be extended. An experimental matrix for bituminous coal will be completed, with particular emphasis on the amount and composition of condensable tars and oils. To include lignite, a brief series of experiments will be completed. With installation of a pressurized block pyrolyzer, effects of pressurized gasification or high-pressure reverse combustion linkage may be evaluated.

## COAL GASIFICATION SIMULATION DATA

SCIENCE APPLICATIONS, INC.

DOE - \$80,945

5/12/77 - Continuing

**OBJECTIVES** – This effort will study, analyze, and interpret data from the Morgantown Energy Research Center's (MERC) Underground Coal Gasification Simulation Laboratory, to identify and

quantify parametric relationships most applicable to a field test, and to recommend the objectives and conditions of future simulation tests.

**RECENT WORK AND ACCOMPLISHMENTS** — A one-dimensional, time-dependent, multispecies, pressure-dependent model applicable to reverse combustion linking, has been synthesized from a constant pressure combustion module and a permeation module. This quasi-steady state model includes mass and energy transfer in both gas and solid phases. Model predictions of flame speed, peak temperatures, pressure effects, heating values, and sensitivity to coal properties fall within the measured data from laboratory tests. Nineteen underground coal gasification simulation tests have been analyzed with emphasis on fourteen tests conducted with bituminous coals. Time domain studies, cross plots, linear regression analysis, and least squares fits were used to correlate the experimental data. Correlations have been determined among input and output parameters as well as between selected output parameters themselves. Time lags in the laboratory system, including physical gas transport and chemical time lags, were specified.

**PLANS FOR THE COMING YEAR** — Upon completion of the analysis and interpretation of the MERC Underground Simulation Laboratory data, recommendations for the objectives and conditions for future simulation tests will be prepared. Using available laboratory and field data, the previously derived model will be refined to facilitate the identification and quantification of parametric relationships applicable to a field test.

## NUMERICAL SIMULATION OF UCG BY STREAM METHOD

PENNSYLVANIA STATE UNIVERSITY  
DOE - \$56,107; Pennsylvania State - \$2,953

**OBJECTIVES** — Work plans include development of two-dimensional (radial and axial, and x-y) models of borehole linking and permeating flow, including the effects of (1) all principal gasification and shift reactions, (2) heat and mass transfer in and adjacent to the burn zone, allowing for flow in the solid as well, (3) oxygen, steam, and carbon dioxide in the injected gas, (4) devolatilization and swelling of coal, and (5) synthesis of the borehole linking and permeating flow models.

**RECENT WORK AND ACCOMPLISHMENTS** — This investigation was devoted to the development of a two-dimensional model of underground coal gasification, via the stream method. The geometry considered consists of an initial horizontal borehole in a coal seam, one end of which is ignited, and air is blown through. Seven reactions, involving carbon, oxygen, carbon dioxide, carbon monoxide, water, hydrogen and methane, are allowed to occur. Nitrogen is the seventh gas component. All reactions, coal, and gas properties are variable functions. As a result of gasification, the channel diameter increases with time, and a combustible gas is produced. The model developed accounts for all these factors. Two basically different approaches were used, and four distinct models were developed in this process. The first model employs a transformation of coordinates which renders the burning coal boundary invariant with time. In spite of the elegance of this approach, the nonlinearities introduced are such that even a direct, simultaneous solution (gas and solid temperatures) necessitates very small time steps. The second approach does not transform the coordinates. The original coordinates are used at all times, but the grid size in the radial direction (perpendicular to air flow) is varied with time, as the coal burns. Simultaneous, direct solution of

the difference equations is employed. This model was tested for nine data sets. The model is extremely complex but runs smoothly. The only limitation is an upper limit on the solid temperature, barring which the temperature will increase to very large values. In other words, a problem should not arise if a realistic carbon oxidation reaction rate equation is employed.

Model 2 was primarily tested for channel dimensions of the type encountered in lab experiments with the idea that it may be employed in MERC's laboratory experiment interpretation. Presumably, it can be utilized for other situations as well. A model having such a degree of complexity requires careful data input, especially as regards the various reaction rates. Unrealistic reaction rates, or demands on the model will give rise to less than perfect results. However, it is believed that the model should be stable under most conditions. Its development is considered a major accomplishment.

**PLANS FOR THE COMING YEAR** – Efficiency of the computer code for Model 2 will be improved. The contract will most probably terminate in March 1978.

## **FLOW PROPERTIES AND CAPILLARY PRESSURE OF UCG COALS**

UNIVERSITY OF PITTSBURGH  
DOE - \$52,014; Pittsburgh - \$11,997  
9/1/76 - 11/30/77

**OBJECTIVES** – Gas-liquid relative permeabilities will be measured and characterized for various subbituminous and bituminous coals as functions of saturation, overburden pressure, and structural orientation. These permeabilities partially define the equations of motion of simultaneous multiphase flow through coal seams. The understanding of the nature of such flows constitutes the principal control for optimum in situ gasification of coal seams to produce a low- or medium-Btu gas for production of electrical power, synthetic natural gas, or chemical feedstocks.

**RECENT WORK AND ACCOMPLISHMENTS** – Numerous relative permeabilities to gas as functions of water saturation have been measured for the Hanna and Gillette, Wyoming subbituminous coals and the Pricetown (Pittsburgh Seam) bituminous coal. All tests were run at a simulated overburden pressure of approximately 200 psi. The three coals displayed significantly different characteristic effective and relative permeabilities to gas. The Pricetown and Gillette coals represent the extreme cases, the Pricetown coal increasing rapidly for small decreases in water saturation and the Gillette coal increasing its permeability to gas gradually to low water saturations, at which point it rises rapidly. The Hanna displays a coupling of both phenomena. Several permeabilities to water as functions of saturation were obtained for the Pricetown and Hanna coals. Porosities and absolute permeabilities of all three coals were measured as functions of simulated overburden pressures over a range of 1500 psi. The property reductions were quite rapid at moderate pressures followed by convergence to exponential behavior, thus allowing extrapolation to higher values of overburden pressure. Several papers have been presented during the contract period.

**PLANS FOR THE COMING YEAR** – A new contract (Et-78-S-02-4639.A000) entitled Measurement of the Flow Properties of Coals for In Situ Gasification has been awarded by DOE for the period ending November 30, 1978. This project will extend the above analysis to other coals of

interest for in situ gasification. These include Texas lignites, steeply dipping coal seams, and coals of other field projects. Also, the effect of overburden on the permeabilities to gas will be investigated over a range of 1000 psi. The effect of CO<sub>2</sub> adsorption will be studied.

## MARKET EVALUATION FOR UCG-PRODUCED GAS

SRI INTERNATIONAL

DOE - \$45,963

8/1/77 - Continuing

**OBJECTIVES** – The contractor is to perform a preliminary market evaluation for gas produced from UCG in the Green River, Fort Union, Powder River, San Juan River, and Denver Basin regions. Forecasts will be prepared for the years 1985 and 2000. Reasons for differences in the marketability of low- and medium-Btu gas will be delineated, and markets for high-Btu gas from UCG will be qualitatively discussed.

**RECENT WORK AND ACCOMPLISHMENTS** – The most important market for low- and medium-Btu gas from UCG is the electric and natural gas utility industry. There may be an urgent need for the electricity so produced. Serious questions about the adequacy of electrical generating capacity in California suggest that satisfaction of electric demand there may require increased transfer of electric power from other states after 1985. Western coal reserves are expected to play a crucial role. Whether direct combustion or underground gasification of western coal is favored for power generation will depend on comparative economics and environmental impacts. Environmental questions surrounding the surface mining and direct combustion of coal may strongly influence prospects for UCG.

**PLANS FOR THE COMING YEAR** – Work yet to be completed includes evaluation of low- and medium-Btu gas costs from UCG relative to alternative energy sources to permit comparisons of overall economics of electric power generation. Industrial markets will be characterized and discussed. The outlook for low- and medium-Btu gas blends with high-Btu gas will be assessed along with prospects for upgrading of the products of UCG to high-Btu gas.

## ELECTROMAGNETIC INSTRUMENTATION IN IN SITU GASIFICATION

WEST VIRGINIA UNIVERSITY

DOE - \$59,996; West Virginia - \$2,838

5/1/76 - 12/31/77

**OBJECTIVES** – This study was to initially investigate and then develop subsurface electromagnetic instrumentation to detect, monitor, and map the burn front of an underground coal conversion process. The progression of the gasification within a coal bed must be monitored and controlled so that significant amounts of coal in the deposits are not bypassed either ungasified or partially gasified. The monitoring technique must have enough resolution to distinctly map the progression of the burning front. The developed electromagnetic instrumentation is to be designed so that it can be accommodated within a subsurface borehole environment and to be used in monitoring field projects such as the one at Pricetown, West Virginia, under the direction of the Morgantown Energy Research Center of the Department of Energy.



**RECENT WORK AND ACCOMPLISHMENTS** – There are three distinct physical planes associated with a coal sample: the bedding (horizontal), the face cleats (vertical), and the butt cleats (vertical) plane. Although the face and butt cleats are both vertical planes, the surface of the face cleats is

much smoother than that of the butt cleats. It has been established by our investigation of many samples of coal that there are marked polarization effects in the conductivity of a solid coal sample. Conductivities lower by a factor of 3 to 6, depending upon the temperature and moisture content of the sample, for each have been measured for each of the following: (1) the electromagnetic wave travels perpendicular to the face cleats and its electric field is parallel to the face and butt cleats but perpendicular to the bedding plane; (2) the electromagnetic wave travels perpendicular to the butt cleats and its electric field is parallel to the butt and face cleats but perpendicular to the bedding plane; and (3) the electromagnetic wave travels perpendicular to the bedding plane and its electric field is parallel to the bedding plane and face cleats but perpendicular to the butt cleats. The polarization phenomenon of coal found in this investigation was one of the factors that led to previous unexplained marked system improvements in electromagnetic transmission within an underground coal seam when the transmitting and receiving loop antennas were oriented in a common vertical plane.

These findings conclude that, because of the polarization phenomenon of coal, the efficiency of an underground electromagnetic system can be improved significantly by properly orienting the transmitting and receiving elements. Using the results of the measurements of the electrical properties of coal, electromagnetic system designs have been undertaken. At present there are two modes of operation for which an electromagnetic system can be used. One mode is the reflection and the other the transmission scheme. A diagram of the conceptual subsurface electromagnetic system is designed to operate in the reflection and transmission modes. Some parts of the system, such as the antennas, have been investigated and tested in the laboratory. In addition the reflection mode system has been assembled in the laboratory and was used to measure unknown lengths of cables.

**PLANS FOR THE COMING YEAR** – A laboratory reflection and transmission mode system will be designed. In-house and procured components will be used to assemble a laboratory reflection and transmission mode system. Computer software will be developed and used to analyze the data collected from the monitoring of the electromagnetic waves of the reflection and transmission mode system.

## **THERMAL AND FLUID-MECHANICAL ASPECTS OF UCG**

WEST VIRGINIA UNIVERSITY  
DOE - \$79,771; West Virginia - \$9,086  
6/1/76 - 8/31/77

**OBJECTIVES** – This study is to develop a one-dimensional mathematical model of the in situ underground coal gasification process. This mathematical model is also to be written into computer program form to solve for the chemical composition of the gasification products and the Btu content of the product gases. The heat losses from the gasification zone to the surroundings and the water intrusion into this zone as well as the chemical kinetics of the gasification process are to be incorporated into the model. Once the UCG process is successfully developed, the energy in coal from seams presently inaccessible or economically unattractive if mined by conventional methods,

can be successfully utilized. Mathematical modeling will aid in the design of UCG field experiments and the interpretation of the resulting field data.

**RECENT WORK AND ACCOMPLISHMENTS** — A one-dimensional mathematical model of the UCG process has been incorporated into a computer program. The model can predict the chemical composition of the gasification products and the Btu content of these gases. The program can perform these calculations as a function of the operating parameters, the water influx into the gasification zone, and the thickness of the coal seam. The effect of the coal seam thickness is a direct function of the heat losses to the surroundings and a separate model of the heat loss mechanism has been integrated into the one-dimensional model. A fourth chemical reaction had to be added to the three reactions generally used in other models to handle cases with low water intrusion rates. The carbon-CO<sub>2</sub> reaction is necessary to describe the reduction process when insufficient water is available for the water-gas reduction process. The program has shown how various parameters such as the flow rate of blast air, the gasification pressure, and the coal seam thickness affect the Btu content of gasification products.

**PLANS FOR THE COMING YEAR** — This contract terminated August 31, 1977.

#### STRUCTURAL MECHANICS SIMULATION OF UCG

WEST VIRGINIA UNIVERSITY  
DOE - \$155,517; West Virginia - \$9,568  
5/1/76 - 4/30/79

**OBJECTIVES** — This research is being undertaken to develop a predictive structural model simulation of the displacements, strains, and stresses associated with in situ coal conversion. This simulation is a satellite of the overall Morgantown Energy Research Center modeling and laboratory program in the areas of reaction kinetics, fluid flow, heat transfer, and structural mechanics for supplementing field testing planned at Pricetown, West Virginia. The analytical results will provide design criteria and fundamental insight on roof collapse, surface subsidence, cavity configuration, flame front stability, and variation of coal permeability.

**RECENT WORK AND ACCOMPLISHMENTS** — The investigations have included preliminary structural formulation and computer simulation of a master model representing the longwall generator (LWG) UCG concept. This finite element model incorporates experimentally determined Pittsburgh coal and adjacent shale overburden mechanical properties for predicting simultaneous roof response and surface subsidence. Supporting studies have been conducted on roof stability and stress analysis of spheroidal and ellipsoidal cavities for the LWG and linked vertical well cavities. Thermo-mechanical crack/channel response studies have been conducted to obtain analytical characterizations of the permeability and fracture response of porous/permeability media. The results are useful in the interpretation of permeability zones, roof cracking, and channel responses during forward and reverse combustion.

**PLANS FOR THE COMING YEAR** — Future investigations will include completion of the finite element master model simulation and associated material property determinations at elevated temperatures. Completion of the permeability, cavity thermo-elastic stress analysis, and crack/channel response models is also envisioned. Work will also be oriented toward supporting of Morgantown Energy Research Center field experiments on underground coal gasification.

## PUBLICATIONS

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## *Abbreviations and Acronyms*

### A

|              |  |
|--------------|--|
| <b>AAG</b>   | agglomerating ash gasifier   |
| <b>AAS</b>   | atomic absorption spectroscopy   |
| <b>ADARS</b> | automatic data logging and data reduction system                                       |
| <b>AE</b>    | Architect and Engineer   |
| <b>AEDC</b>  | Arnold Engineering Development Center  |
| <b>AERL</b>  | AVCO Everett Research Laboratory Inc.  |
| <b>AES</b>   | atomic emission spectroscopy, Auger electron spectrometer, automated extraction system |
| <b>AFBC</b>  | atmospheric fluidized-bed combustion   |
| <b>AGA</b>   | American Gas Association   |
| <b>amp</b>   | ampere   |
| <b>AMRC</b>  | Albany Metallurgy Research Center  |
| <b>ANL</b>   | Argonne National Laboratory  |
| <b>API</b>   | American Petroleum Institute   |
| <b>ART</b>   | applicable Russian technology  |
| <b>ASTM</b>  | American Society for Testing Materials   |
| <b>atm</b>   | atmospheric (of pressure)  |
| <b>atma</b>  | atmosphere absolute  |
| <b>ATP</b>   | adenosene triphosphate   |

### B

|               |                                     |
|---------------|-------------------------------------|
| <b>BA</b>     | Budget Authority                    |
| <b>bbl</b>    | barrel                              |
| <b>bbl/d</b>  | barrels per day                     |
| <b>bbl/sd</b> | barrels per stream day              |
| <b>BCA</b>    | benzene carboxylic acid             |
| <b>BCR</b>    | Bituminous Coal Research, Inc.      |
| <b>BERC</b>   | Bartlesville Energy Research Center |
| <b>BHTP</b>   | bottom-hole treating pressure       |
| <b>BNL</b>    | Brookhaven National Laboratory      |

|                |                              |
|----------------|------------------------------|
| <b>BO</b>      | Budget Outlay                |
| <b>BOD</b>     | biochemical oxygen demand    |
| <b>BOM</b>     | Bureau of Mines              |
| <b>BSB</b>     | blind shaft borer            |
| <b>Btu</b>     | British thermal unit         |
| <b>BTX</b>     | benzene, toluene, and xylene |
| <b>B&amp;W</b> | Babcock and Wilcox           |

### C

|                          |  |
|--------------------------|--|
| <b>CAPD</b>              | combustion and advanced power development        |
| <b>CBC</b>               | carbon burnup cell                               |
| <b>CBFDP</b>             | clean boiler fuel demonstration plant            |
| <b>CC</b>                | coal conversion                                  |
| <b>CCP</b>               | coal conversion processes                        |
| <b>CDIF</b>              | Component Development and Integration Facility   |
| <b>CDS</b>               | Chemical Data Systems                            |
| <b>CEF</b>               | chemical explosive fracturing                    |
| <b>CEI</b>               | Combustion Engineering, Inc.                     |
| <b>CFCC</b>              | coal-fired combined cycle                        |
| <b>CFFC</b>              | clean fuel from coal                             |
| <b>CGA</b>               | coal gasification atmospheres                    |
| <b>CGCC</b>              | coal gasification combined cycle                 |
| <b>C-h</b>               | candle-hour                                      |
| <b>cm</b>                | centimeter                                       |
| <b>CO<sub>2</sub></b>    | carbon dioxide                                   |
| <b>CO-H<sub>2</sub>O</b> | carbon monoxide-water                            |
| <b>COD</b>               | chemical oxygen demand                           |
| <b>COE</b>               | crude oil equivalent                             |
| <b>COED</b>              | coal oil energy development—liquefaction process |
| <b>COG</b>               | coal-oil-gas                                     |

|                  |  |                       |  |
|------------------|--|-----------------------|--|
| <b>COM</b>       | coal-oil mixture                               | <b>EIA</b>            | Environmental Impact Assessment              |
| <b>CONBEC</b>    | connected-block effective conductivity         | <b>EIS</b>            | Environmental Impact Statement               |
| <b>Conoco</b>    | Continental Oil Co.                            | <b>EOR</b>            | enhanced oil recovery                        |
| <b>cp</b>        | centipoise                                     | <b>EPA</b>            | Environmental Protection Agency              |
| <b>CPU</b>       | Combustion Power Unit                          | <b>ERC</b>            | Energy Research Center                       |
| <b>CTF</b>       | Combustion Test Facility                       | <b>ER&amp;E</b>       | Exxon Research and Engineering Company       |
| <b>CTIF</b>      | Component Test and Integration Facility        | <b>ERTS</b>           | Earth Resources Technology Satellite         |
| <b>CTIU</b>      | Component and Test Integration Unit            | <b>ESR</b>            | electron spin resonance                      |
| <b>CUNY</b>      | City University of New York                    | <b>ETF</b>            | Engineering Test Facility                    |
| <b>CW</b>        | Curtiss-Wright                                 | <b>F</b>              |  |
| <b>CY</b>        | Calendar Year                                  | <b>F</b>              | faraday                                      |
| <b>D</b>         |  | <b>°F</b>             | degrees Fahrenheit                           |
| <b>db</b>        | decibel  | <b>FBC</b>            | fluidized-bed combustion                     |
| <b>DDP</b>       | Detailed Development Plan                      | <b>FBNML</b>          | Francis Bitter National Magnet Laboratory    |
| <b>DE&amp;OT</b> | Drilling, Exploration, and Offshore Technology | <b>FDP</b>            | free-fall dilute phase                       |
| <b>DOC</b>       | Department of Commerce                         | <b>FE</b>             | Fossil Energy                                |
| <b>DOD</b>       | Department of Defense                          | <b>FEARB</b>          | Fossil Energy Authorization and Review Board |
| <b>DOE</b>       | Department of Energy                           | <b>FGD</b>            | flue gas desulfurization                     |
| <b>DOI</b>       | Department of Interior                         | <b>FIMS</b>           | field ionization mass spectrometry           |
| <b>DSC</b>       | differential scanning calorimetry              | <b>FMA</b>            | Foster-Miller Associates Inc.                |
| <b>DTA</b>       | differential thermal analysis                  | <b>ft</b>             | foot, feet                                   |
| <b>DWT</b>       | deviated well test                             | <b>ft<sup>3</sup></b> | cubic feet                                   |
| <b>E</b>         |  | <b>ft/sec</b>         | feet per second                              |
| <b>EA</b>        | Environmental Assessment                       | <b>FWEC</b>           | Foster Wheeler Energy Corporation            |
| <b>ECAS</b>      | Energy Conversion Alternatives Study           | <b>FY</b>             | Fiscal Year                                  |
| <b>ECP</b>       | Engineering Change Proposal                    | <b>G</b>              |  |
| <b>EDP</b>       | Environmental Development Plan                 | <b>GC</b>             | gas chromatography                           |
| <b>EDS</b>       | Exxon Donor Solvent                            | <b>GE</b>             | General Electric Company                     |
| <b>EFB</b>       | electrofluidized bed                           | <b>GFERC</b>          | Grand Forks Energy Research Center           |
| <b>EGR</b>       | enhanced gas recovery                          | <b>GPC</b>            | gel permeation                               |
| <b>EGSP</b>      | Eastern Gas Shales Project                     | <b>gpm</b>            | gallons per minute                           |
| <b>EH&amp;S</b>  | Environmental Health & Safety                  | <b>gr</b>             | gram   |

|             |                                       |
|-------------|---------------------------------------|
| <b>GRI</b>  | Gas Research Institute                |
| <b>GSU</b>  | Gulf States Utilities                 |
| <b>GTF</b>  | Generator Test Facility               |
| <b>GURC</b> | Gulf Universities Research Consortium |

## H

|                       |  |
|-----------------------|--|
| <b>H-Coal</b>         | hydrogenated coal                        |
| <b>HDS</b>            | hydrodesulfurization                     |
| <b>HGI</b>            | Hardgrove Grindability Index             |
| <b>HGMS</b>           | high-gradient magnetic separator         |
| <b>HGR</b>            | hot gas recycle                          |
| <b>HHV</b>            | high heating value                       |
| <b>HMF</b>            | high mass flux                           |
| <b>hp</b>             | horsepower                               |
| <b>HPDE</b>           | high-performance demonstration equipment |
| <b>HRI</b>            | Hydrocarbon Research Inc.                |
| <b>H<sub>2</sub>S</b> | hydrogen sulfide                         |
| <b>HTHP</b>           | high temperature, high pressure          |
| <b>HTTT</b>           | high-temperature turbine technology      |
| <b>HVAB</b>           | high volatile A bituminous coal          |

## I

|                 |   |
|-----------------|---|
| <b>IBG</b>      | intermediate-Btu gas                              |
| <b>IC</b>       | internal combustion                               |
| <b>ICAP</b>     | induction coupled argon plasma                    |
| <b>ICGG</b>     | Illinois Coal Gasification Group                  |
| <b>I.D.</b>     | inside diameter                                   |
| <b>IGT</b>      | Institute of Gas Technology                       |
| <b>IITRI</b>    | IIT Research Institute                            |
| <b>IR&amp;T</b> | International Research and Technology Corporation |
| <b>ISIP</b>     | instantaneous shutin pressure                     |
| <b>IVTAN</b>    | Institute of High Temperatures (Soviet Union)     |

## J

|              |                             |
|--------------|-----------------------------|
| <b>JANAF</b> | Joint Army, Navy, Air Force |
|--------------|-----------------------------|

## K

|            |           |
|------------|-----------|
| <b>kg</b>  | kilogram  |
| <b>kHz</b> | kilohertz |
| <b>kv</b>  | kilovolt  |
| <b>kw</b>  | kilowatt  |

## L

|                       |                                   |
|-----------------------|-----------------------------------|
| <b>LASL</b>           | Los Alamos Scientific Laboratory  |
| <b>lb</b>             | pound                             |
| <b>LBG</b>            | low-Btu gas                       |
| <b>LBL</b>            | Lawrence Berkeley Laboratory      |
| <b>lbm</b>            | pound-mass                        |
| <b>LERC</b>           | Laramie Energy Research Center    |
| <b>LLL</b>            | Lawrence Livermore Laboratory     |
| <b>LMMD</b>           | liquid-metal magnetohydrodynamics |
| <b>LN<sub>2</sub></b> | liquid nitrogen                   |
| <b>LNG</b>            | liquefied natural gas             |
| <b>LPG</b>            | liquefied petroleum gas           |
| <b>LPM</b>            | liquid-phase methanation          |
| <b>LRSD</b>           | long-range strategy division      |
| <b>LVW</b>            | linked vertical well              |
| <b>LWG</b>            | longwall generator                |

## M

|                   |   |
|-------------------|---|
| <b>M</b>          | thousand  |
| <b>MAF</b>        | moisture and ash free                             |
| <b>MCA</b>        | Magnetic Corporation of America                   |
| <b>Mcf</b>        | thousand cubic feet                               |
| <b>Mcf/d</b>      | thousand cubic feet per day                       |
| <b>MFCD</b>       | Multiple-Face Continuous Drivage                  |
| <b>MCMS&amp;T</b> | Montana College of Mineral Science and Technology |
| <b>md</b>         | millidarcy  |
| <b>MERC</b>       | Morgantown Energy Research Center                 |



|                       |   |               |   |
|-----------------------|---|---------------|---|
| <b>MERDI</b>          | Montana Energy and MHD Research and Development Institute, Inc. | <b>NSF</b>    | National Science Foundation                   |
| <b>MESA</b>           | Mining Enforcement and Safety Administration                    | <b>NSPS</b>   | New Source Performance Standards              |
| <b>MFB</b>            | multicell fluidized-bed boiler                                  | <b>NTIS</b>   | National Technical Information Service        |
| <b>O</b>              |   |               |   |
| <b>MFCD</b>           | multiple face continuous drivage                                | <b>OCMHD</b>  | open-cycle magnetohydrodynamics               |
| <b>MHD</b>            | magnetohydrodynamics  | <b>OCR</b>    | Office of Coal Research                       |
| <b>MHF</b>            | Massive Hydraulic Fracturing                                    | <b>OCS</b>    | Outer Continental Shelf                       |
| <b>MIT</b>            | Massachusetts Institute of Technology                           | <b>O.D.</b>   | outside diameter                              |
| <b>MIUS</b>           | Modular Integrated Utility Systems                              | <b>ODE</b>    | ordinary differential equation                |
| <b>MLWG</b>           | Memphis Light, Water, and Gas Division                          | <b>ONR</b>    | Office of Naval Research                      |
| <b>MM</b>             | million   | <b>OPDD</b>   | Overall Plant Design Description              |
| <b>MMbbl/d</b>        | millions of barrels per day                                     | <b>OPEC</b>   | oil producing and exporting countries         |
| <b>MPF</b>            | micellar polymer flooding                                       | <b>OPPA</b>   | Office of Program Planning and Analysis       |
| <b>MPPM</b>           | Materials-Process-Product Model                                 | <b>ORC</b>    | Occidental Research Corporation               |
| <b>MS</b>             | mass spectral, mass spectroscopy                                | <b>ORNL</b>   | Oak Ridge National Laboratory                 |
| <b>MSCG</b>           | molten-salt coal gasification                                   | <b>OSHA</b>   | Occupational Safety and Health Administration |
| <b>MSP</b>            | marine sediment penetrators                                     | <b>OS/IES</b> | on-site/integrated energy systems             |
| <b>MSTF</b>           | Molten-Salt Test Facility                                       | <b>OSU</b>    | Oklahoma State University                     |
| <b>MSU</b>            | Montana State University  | <b>P</b>      |   |
| <b>MSW</b>            | municipal solid waste   | <b>PAD</b>    | Program Approval Document                     |
| <b>Mw</b>             | megawatt  | <b>PAH</b>    | polynuclear aromatic hydrocarbon              |
| <b>Mw<sub>e</sub></b> | megawatt electrical   | <b>PB</b>     | packed bed                                    |
| <b>MY</b>             | man-year  | <b>PBP</b>    | packed bed process                            |
| <b>N</b>              |   |               |   |
| <b>NaCl</b>           | sodium chloride   | <b>PCE</b>    | plant and capital equipment                   |
| <b>NCB</b>            | National Coal Board (England)                                   | <b>PCOB</b>   | powdered coal-oil blends                      |
| <b>NEPA</b>           | National Environmental Policy Act                               | <b>PCWS</b>   | primary cooling water system                  |
| <b>NIOSH</b>          | National Institute of Occupational Safety and Health            | <b>Pd</b>     | palladium                                     |
| <b>NL</b>             | National Laboratory   | <b>PDE</b>    | partial differential equation                 |
| <b>NMR</b>            | nuclear magnetic resonance                                      | <b>PDU</b>    | Process Development Unit                      |
| <b>NO<sub>x</sub></b> | nitrogen oxides   | <b>PEDU</b>   | Process and Equipment Development Unit        |
| <b>NPDES</b>          | National Pollutant Discharge Elimination System                 | <b>PER</b>    | Pope, Evans and Robbins                       |
|                       |   | <b>PERC</b>   | Pittsburgh Energy Research Center             |

|                  |  |                       |  |
|------------------|--|-----------------------|--|
| <b>PFB</b>       | pressurized fluidized bed  | <b>scf/h</b>          | standard cubic feet per hour                 |
| <b>PFBC</b>      | pressurized fluidized-bed combustion   | <b>scf/m</b>          | standard cubic feet per minute               |
| <b>PI</b>        | petrochemical intermediates  | <b>SCMS</b>           | superconducting magnet system                |
| <b>PNA</b>       | polynuclear aromatic compounds   | <b>SCOOP</b>          | Stage Combustion Operation Optimizer Program |
| <b>PNG</b>       | petroleum and natural gas  | <b>SCPE</b>           | subblend pairing with pillar extraction      |
| <b>POGO</b>      | Power-Oil-Gas and Other  | <b>sd</b>             | stream day                                   |
| <b>PON</b>       | Program Opportunity Notice   | <b>SDB</b>            | steeply dipping bed                          |
| <b>PPD</b>       | Project Plan Document  | <b>SDD</b>            | System Design Description                    |
| <b>PPHTS</b>     | particle precipitating heat transfer service                                 | <b>SDL</b>            | Spectron Development Laboratories            |
| <b>ppm</b>       | parts per million  | <b>SAEM</b>           | scanning and analyzing electron microscope   |
| <b>PRD</b>       | prime reference design   | <b>SEB</b>            | Source Evaluation Board                      |
| <b>psi</b>       | pounds per square inch   | <b>SEM</b>            | Scanning Electron Microscopy                 |
| <b>psia</b>      | pounds per square inch absolute  | <b>SFME</b>           | single-face multiple-entry                   |
| <b>psig</b>      | pounds per square inch gauge   | <b>SL</b>             | Sandia Laboratory                            |
| <b>PV</b>        | pore volume  | <b>SLAT</b>           | standard liquefaction activity test          |
| <b>Q</b>         |  | <b>SO<sub>2</sub></b> | sulfur dioxide                               |
| <b>Quad</b>      | 10 <sup>15</sup> (quadrillion) Btu's   | <b>SO<sub>3</sub></b> | sulfur trioxide                              |
| <b>R</b>         |  | <b>SO<sub>x</sub></b> | oxides of sulfur                             |
| <b>RBR</b>       | recirculating bed reactor  | <b>SNG</b>            | substitute natural gas                       |
| <b>R&amp;D</b>   | Research and Development   | <b>S-R</b>            | Stearns-Roger Engineering Co.                |
| <b>RD&amp;D</b>  | Research, Development, and Demonstration                                     | <b>SRC</b>            | solvent refined coal                         |
| <b>RDD&amp;C</b> | Research, Development, Demonstration, and Commercialization                  | <b>SRI</b>            | SRI International                            |
| <b>RF</b>        | radio frequency  | <b>SRL</b>            | solvent refined lignite                      |
| <b>RESPONS</b>   | Regional Energy System for the Planning & Optimization of National Scenarios | <b>SRSA</b>           | Special Research Support Agreement           |
| <b>RFP</b>       | Request for Proposal   | <b>Syngas</b>         | synthetic natural gas                        |
| <b>RMC</b>       | Reynolds Metals Company  | <b>T</b>              |  |
| <b>RMP</b>       | Ralph M. Parsons Co.   | <b>T</b>              | tesla  |
| <b>ROI</b>       | Return on Investment   | <b>TBM</b>            | Tunnel Boring Machine                        |
| <b>S</b>         |  | <b>Tcf</b>            | trillion cubic feet                          |
| <b>SARA</b>      | Solvents-Aromatics-Resins-Asphaltenes  | <b>t/d</b>            | tons per day                                 |
| <b>scf</b>       | standard cubic feet  | <b>TDS</b>            | total dissolved solids                       |
| <b>scf/d</b>     | standard cubic feet per day  | <b>TGA</b>            | thermogravimetric analysis                   |

---

|             |   |                 |   |
|-------------|---|-----------------|---|
| <b>TOC</b>  | total organic carbon                    | <b>USGS</b>     | United States Geological Survey         |
| <b>TR</b>   | thermal recovery                        | <b>USS</b>      | United States Steel                     |
| <b>TRV</b>  | technology readiness vehicle            | <b>U.S.S.R.</b> | Union of Soviet Socialist Republics     |
| <b>TSE</b>  | test support equipment                  | <b>UTSI</b>     | University of Tennessee Space Institute |
| <b>TSG</b>  | Technology Support Group                |                 | <b>W</b>                                |
| <b>TTU</b>  | Technology Test Unit                    | <b>w/o</b>      | weight percent                          |
|             | <b>U</b>                                | <b>WOR</b>      | water-to-oil ratio                      |
| <b>UCG</b>  | underground coal gasification           | <b>WRC</b>      | Water Resources Council                 |
| <b>U.K.</b> | United Kingdom                          |                 | <b>X</b>                                |
| <b>USBM</b> | United States Bureau of Mines           | <b>XPS</b>      | X-ray photoelectron spectroscopy        |
| <b>USDA</b> | United States Department of Agriculture | <b>XRD</b>      | X-ray diffraction                       |

## Glossary

The intent of this glossary is to give a general definition of terminology as used in this report. A glossary is considered desirable because of the diverse origin of the technology and broad spectrum of potential readers. For more precise and detailed definitions, the reader is referred to *The Annual Book of ASTM Standards* published by the American Society for Testing Materials (ASTM), *Chemical Engineers' Handbook* by R.H. Perry and C.H. Chilton, and *A Dictionary of Mining, Mineral, and Related Terms* published in 1968 by the U.S. Department of the Interior.

**Å** – Angstrom unit, a unit of length equal to  $10^{-10}$  meters or  $10^{-4}$  microns, generally used as the unit for describing interatomic distances; as an example, the carbon atoms in diamond are 1.5 Å apart.

**absorption** – the dissolution of a gas in a liquid.

**acceptors** – calcined carbonates that absorb carbon dioxide evolved during gasification, exothermically.

**acid-gas removal** – a section of a gas plant where hydrogen sulfide and carbon dioxide are removed from the gas stream.

**activated carbon** – carbon obtained by carbonization in the absence of air, preferably in a vacuum; has the property of absorbing large quantities of gases, solvent vapors; used also for clarifying liquids.

**adiabatic** – any process where heat is neither given off nor absorbed.

**adsorption** – the physical and chemical adherence of a gas to the surface of a solid.

**agglomerate** – assemblage of ash particles rigidly joined together, as by partial fusion (sintering).

**anode carbon** – carbon of high purity, usually crystallized to graphite form, widely used in Leclanche cells, in rods for alumina refining, in electric arcs and nuclear reactors.

**anthracite coal** – any coal containing 86 to 98 percent fixed carbon, on a dry, mineral-matter-free basis.

**aromatic hydrocarbon** – an unsaturated cyclic hydrocarbon containing one or more six-carbon rings.

**ash** – theoretically, the inorganic salts contained in coal; practically, the residue from the combustion of dried coal that has been burned at 1,380°F.

**autoclave** – a vessel, constructed of thick-walled steel (alloy steel or nickel alloys), for carrying out chemical reactions under pressure and at high temperatures.

**bench-scale unit** – a small-scale laboratory unit for testing process concepts and operating criteria as a first step in the evaluation of a process.

**binder** – carbon products, tars, etc., used to impart cohesion to the body to be formed; a coal-extract binder may be used to prepare formed-coke pellets from noncoking coals.

**bituminous coal** – a broad class of coals containing 46 to 86 percent fixed carbon and 20 to 40 percent volatile matter.

**blow down** – the removal of liquids from a process vessel by the application of pressure.

**bottoming cycle** – the lower temperature thermodynamic power cycle of a combined-cycle system.

**Btu** – British thermal unit, the quantity of energy required to raise the temperature of 1 lb of water 1°F.

**BTX** – benzene, toluene, xylene; aromatic hydrocarbons; toluene is methyl-benzene, xylene is dimethyl benzene.

**caking** – the softening and agglomeration of coal as a result of the application of heat.

**calcine** – to heat a solid to a high temperature to cause the decomposition of hydrates and carbonates.

**carbon fiber** – very fine filaments about 8 microns in diameter that are used in composite materials, being bound with resins.

**carbonization** – the destructive distillation of coal in the absence of air accompanied by the formation of char (coke), liquid (tar), and gaseous products.

**catalyst** – a substance that accelerates the rate of a chemical reaction without itself undergoing a permanent chemical change.

**centrifuge** – an apparatus rotating at high speed to separate solids from liquids, e.g., undissolved residue from coal solution in the SRC process.

**char** – the solid residue from coal after the removal of moisture and volatile matter, i.e., essentially ash plus fixed carbon.

**closed cycle** – a thermodynamic power cycle in which the working fluid is recycled.

**coal** – a natural solid material consisting of amorphous elemental carbon with various amounts of organic and inorganic compounds.

**coke** – a solid consisting primarily of amorphous carbon having certain properties of strength, cell structure, and minimum impurities, and manufactured by the thermal decomposition of petroleum residues and certain types of coal.

**coke breeze** – coke particles smaller than 1/2 inch.

**combined cycle** – two sequential thermodynamic power conversion systems operating at different temperatures.

**combustion gas** – gas formed by the rapid oxidation of coal, e.g., burning.

**combustor** – a vessel in which combustion of gaseous products from a fuel takes place by the chemical union of oxygen with the gas.

**coupon** – a polished metal strip used to measure the rate of corrosion of the metal in a specific gaseous or liquid environment.

**cracking** – the partial decomposition of high-molecular-weight organic compounds into lower-molecular-weight compounds, generally as a result of high temperatures.

**crude gas** – gas produced in a gasifier containing a wide range of impurities, also known as offgas.

**cyclone separator** – essentially a settling chamber to separate solid particles from a gas, in which gravitational acceleration is replaced by centrifugal acceleration.

- degasification** – a process for draining naturally occurring methane from coal seams.
- delayed coking** – a process whereby coal is subjected to a long period of carbonization at moderate temperatures to form metallurgical coke.
- demineralization** – removal of mineral matter (ash) from coal by solvent extraction, usually under hydrogen atmosphere.
- demonstration plant** – a plant whose design is based on data derived from pilot-scale testing of sufficient capacity to demonstrate the large-scale feasibility of a process.
- depolymerization** – the change of a large molecule (e.g., coal polymers) into simpler molecules (e.g., aromatics, BTX), usually accompanied by the substitution of hydrogen for oxygen.
- destructive distillation** – the distillation of coal or other solids accompanied by their decomposition; destructive distillation of coal yields coke, tar, ammonia, gas, etc.
- desulfurization** – the removal of sulfur from hydrocarbonaceous substances by chemical reactions. Various processes are Claus, Appleby-Frodingham, C.S.I.R.O., ferric chloride leaching, Kennecott.
- devolatilization** – the removal of a proportion of the volatile matter from medium- and high-volatile coals to prevent subsequent caking.
- dissolution** – the taking up of a substance by a liquid with the formation of a homogeneous solution.
- distillation** – a process of evaporation and recondensation used for separating liquids into various fractions according to their boiling points or boiling ranges.
- dolomite** – a mineral having the chemical formula  $\text{CaMg}(\text{CO}_3)_2$ , i.e., a carbonate of calcium and magnesium.
- ebullated bed** – a boiling bed; gas, containing a relatively small proportion of suspended solids, bubbles through a higher-density fluidized phase, with the result that the system resembles a boiling liquid.
- economizer** – a heat exchanger for recovering heat from flue gases and using it to heat feedwater or combustion air.
- effluent gas** – gas issuing from a gasifier or combustor.
- electrode carbon** – see anode carbon.
- elutriation** – the preferential removal of the small constituents of a mixture of solid particles by a stream of high-velocity gas.
- endothermic** – a process in which heat is absorbed.
- enthalpy** – a form of thermal energy defined as the sum of the internal energy of a system plus the product of the system's volume and pressure.
- entrained bed** – a bed in which solid particles are suspended in a moving fluid and are progressively carried over in the effluent stream.
- entrained flow** – see entrained bed.
- eutectic** – that combination of two or more components which produces the lowest melting temperature.
- exothermic** – a process in which heat is liberated.
- extraction** – a process for dissolving certain constituents of a mixture by means of a liquid with solvent properties for selected components only.
- extraction-hydrogenation** – extraction carried out in the presence of hydrogen either as a gas or derived by transfer from hydrogen donor solvents.
- extractive coking** – similar to delayed coking process, with the emphasis on high tar yields to produce liquids.
- filter cake** – the moist residue remaining from the filtration of a slurry to produce a clean filtrate.
- filtrate** – a liquid free of solid matter after having passed through a filter.
- filtration** – the separation of solids from liquids by passing the mixture through a suitable medium, e.g., cloth, paper, diatomite.
- Fischer assay** – an assay for the determination of oil (tar) yields from coal or oil shale; conducted in a retort under an inert atmosphere with a gradual increase in temperature.
- Fischer-Tropsch catalyst** – iron and cobalt catalysts developed by Fischer and Tropsch for the catalytic synthesis of liquid fuels from coal-derived synthesis gas.
- fixed bed** – solid particles in intimate contact with fluid passing through them, but too slowly to cause fluidization.
- fixed carbon** – theoretically, the carbon content of coal which exists in the elemental state; practically, the difference between 100 percent and the sum of ash, moisture, and volatile matter percentages.
- flash carbonization** – a carbonization process characterized by very short residence times of coal in the reactor to optimize tar yields; also called flash pyrolysis.
- flue gas** – gas issuing from a combustor; either exhausted to atmosphere or expanded through a gas turbine.
- fluidization (dense phase)** – the turbulent motion of solid particles in a fluid stream; the particles are close enough to interact and give the appearance of a boiling liquid.
- fluidization (entrained)** – solid particles transported by a high-velocity fluid stream with little or no solid interaction.
- fluidized bed** – a bed through which a fluid is passed with a velocity high enough for the solid particles to separate and become freely supported in the fluid.
- fly-ash** – a fine ash from the pulverized coal burned in power station boilers, or entrained ash carried over from a gasifier.
- fractionation** – distillation process for the separation of the various components of liquid mixtures; an effective separation can only be achieved by the use of fractionating columns attached to the still; also called fractional distillation.
- fuel cell** – a galvanic cell in which the oxidation of a fuel (e.g., coal) is utilized to produce electricity.
- fuel gas** – low heating value product generally utilized on-site for power generation or industrial use.
- gasification of coal** – the conversion of solid coal into a gaseous form by any of a variety of chemical processes.
- gasifier** – a vessel in which gasification occurs, usually utilizing fixed-bed, fluidized-bed, or entrained-bed units.
- high-Btu gas** – a gas, largely methane, having a heating value of 900 to 1,000 Btu per cubic foot, which approaches the value for natural gas.
- high heating value (HHV)** – the heat liberated during a combustion process in which the product water vapor is condensed to a liquid.
- hydrocoking** – coking of tars, SRC, etc., under hydrogenating conditions to form liquid products.
- hydrocracking** – the combination of cracking and hydrogenation of organic compounds.

- hydrogasification** – gasification that involves the addition of hydrogen to the products of primary gasification to optimize formation of methane.
- hydrogenation** – chemical reactions involving the addition of hydrogen, present as a gas, to a substance in the presence of a catalyst under high temperatures and pressures.
- hydrogen donor solvent** – solvent, such as anthracene oil, tetralin (tetrahydronaphthalene), decalin, etc., which transfers hydrogen to coal constituents causing depolymerization and consequent evolution of liquid products of lower boiling range which are taken up by the solvent.
- hydrotreating** – a process involving the reaction of hydrogen with hydrocarbon mixtures for the removal of such impurities as oxygen, nitrogen, and sulfur.
- ignition temperature** – the minimum temperature necessary to initiate self-sustained combustion of a substance.
- industrial gas** – see fuel gas.
- inerts** – macerals in coal not readily changed by the action of solvents in the solvent extraction of coal, e.g., fusinite.
- in situ** – in its original place, e.g., underground gasification of a coal seam.
- intermediate-Btu (IBtu) gas** – synthesis gas product with an HHV between 250 and 500 Btu per standard cubic foot, consisting mainly of carbon monoxide and hydrogen.
- kerogen** – organic material from which shale oil is extracted.
- lignite** – a low rank of coal between peat and subbituminous.
- limestone** – a sedimentary rock composed mostly of calcium carbonate ( $\text{CaCO}_3$ ) and possibly some magnesium carbonate ( $\text{MgCO}_3$ ).
- liquefaction** – conversion of a solid to a liquid; with coal this invariably involves hydrogenation to depolymerize the coal molecules to simpler molecules.
- liquified petroleum gas (LPG)** – those hydrocarbons that have a vapor pressure (at 70°F) slightly above atmospheric (such as propane and butane); kept in liquid form under a pressure higher than 1 atm.
- lock hopper** – a mechanical device that permits the introduction of a solid into an environment of different pressure.
- low-Btu (LBtu) gas** – a gas having a heating value of 150 to 350 Btu per cubic foot; when made from coal, water, and air, it contains varying quantities of carbon monoxide, carbon dioxide, nitrogen, hydrogen, and methane.
- moisture and ash-free (MAF)** – a term that relates to the organic fraction in coal; “moisture- and mineral-matter free” is equivalent.
- methanation** – the production of methane ( $\text{CH}_4$ ) from a mixture of carbon monoxide and hydrogen.
- micron** – a unit of length equal to 1 millionth of a meter.
- natural gas** – naturally occurring gas extracted from sedimentary structures consisting mainly of methane and having an HHV to 1050 Btu per standard cubic foot.
- noncoking** – a coal that does not form coke under normal coking conditions.
- olefinic hydrocarbon** – a class of unsaturated hydrocarbons containing one or more double bonds and having the general chemical formula  $\text{C}_n\text{H}_{2n}$ .
- open cycle** – a thermodynamic power cycle in which the working fluid passes through the system only once and is then exhausted to the atmosphere.
- perfect gas** – see ideal gas.
- petrochemicals** – those derived from crude oil or natural gas, or their coal-derived substitutes; they include light hydrocarbons such as butylene, ethylene, and propylene—the raw materials for the production of plastics by polymerization.
- phenols** – a group of aromatic compounds having the hydroxyl group directly attached to the benzene ring. They give the reactions of alcohols, forming esters, ethers, and thiocompounds; phenols are more reactive than the benzene hydrocarbons; derived from coal tar.
- pilot plant** – a chemical process plant containing all the processes of a commercial unit, but on a smaller scale, for the purpose of studying the process.
- pipeline gas** – a methane-rich gas that conforms to certain standards and having an HHV between 950 and 1,050 Btu per standard cubic foot. Standards include minimum water content, minimum inert gases, minimum hydrogen and carbon monoxide content, and its compressibility to 1,000 psig.
- process development unit** – a laboratory-sized system used to study the effects of process variables on performance.
- proximate analysis** – analysis of coal based on the percentages of moisture, volatile matter, fixed carbon, and ash.
- purification** – removal of the wide range of impurities present in gases from coal gasification to yield purity gas. See Rectisol process.
- pyrolysis** – thermal decomposition of organic compounds in the absence of oxygen.
- quenching** – cooling by immersion in oil or water bath or spray.
- Raney nickel catalyst** – nickel sponge used as a catalyst in the hydrogenation of organic materials and the methanation of synthesis gas to methane.
- raw gas** – see crude gas
- reactivity** – susceptibility to chemical change; in coal conversion, the reactivity of the coal for conversion to liquid products is a function of the MAF volatile matter content and the petrographic composition of the coal.
- reactor** – vessel in which coal-conversion reactions take place.
- reducing gas** – used as a reducing agent in redox reactions, e.g., hydrogen, superheated steam.
- reforming processes** – a group of proprietary processes in which low-grade or low molecular weight hydrocarbons are catalytically reformed to higher grade or higher molecular weight materials; also applies to the endothermic reforming of methane, for the production of hydrogen by the reaction of methane and steam in the presence of nickel catalysts.
- refractory** – a material capable of withstanding extremely high temperatures and having relatively low thermal conductivities.

residence time – time spent by a typical particle in a reaction zone.

retort – distill or decompose by heat.

saturated hydrocarbon – a hydrocarbon in which all bonds are single covalent bonds and none are double or triple bonds.

scrubber – apparatus in which a gas stream is freed of tar, ammonia, and hydrogen sulfide.

semi-water gas – a mixture of carbon monoxide, carbon dioxide, hydrogen, and nitrogen obtained by passing an air-stream mixture through an incandescent bed of coke; HHV about 120 Btu per standard cubic foot.

sensible heat – that heat which results in only the elevation of the temperature of a substance with no phase changes.

shift conversion – process for the production of gas with a desired carbon monoxide content from crude gases derived from coal gasification; carbon monoxide rich gas is saturated with steam and passed through a catalytic reactor where the carbon monoxide reacts with steam to produce hydrogen and carbon dioxide, the latter being subsequently removed in a wash plant. The ratio of hydrogen to carbon monoxide in the product gas can be changed at will.

sintering – the agglomeration of solids at temperatures below their melting point, usually as a consequence of heat and pressure.

slag – a molten mixture of various metallic oxides and salts.

slurry – a suspension of pulverized solid in a liquid.

solvation – the association or combination of molecules of solvent with solute ions or molecules.

solvent – that component of a solution which is present in excess, or whose physical state is the same as that of the solution.

solvent extraction – selective transfer of desired coal constituents from finely divided coal particles into a suitable solvent after intimate mixing, usually at high temperatures and pressures in the presence of hydrogen, with or without a catalyst, followed by phase separation.

solvent refined coal (SRC) – a coal extract derived by solvent extraction; a brittle, vitreous solid (M.P. 300°F to 400°F) containing about 0.1 percent ash and about 10 percent of the sulfur in the original coal feedstock; calorific value is about 16,000 Btu per pound. May be used as a clean fuel for power generation by combustion; utilized for the production of high-grade metallurgical coke, anode carbon, and activated carbon by coking; or hydrogenated to produce synthetic crude oil.

space velocity – the volume of a fluid (usually measured at standard conditions) passing through a unit volume in a unit time; units are in reciprocal time.

spectroscopic – spectral or pertaining to the optic spectrum.

standard cubic foot (scf) – the volume of a gas at standard conditions of temperature and pressure. The American

Gas Association uses moisture-free gas at 60°F and 30 inches of mercury (1.0037 atm) as its standard conditions. The pressure standard is not universal in the gas industry; 14.7 psia (1.000 atm) and 14.4 psia (0.980 atm) are also used. The scientific community uses 32°F and 1 atm as standard conditions.

stoichiometry – the definite proportions in which molecules react chemically to form new molecules.

stripping – the removal of the more volatile components from a liquid mixture of compounds.

subbituminous coal – the rank of coal between bituminous and lignite, classified by ASTM as having a range of heating values between 8,300 and 11,000 Btu per pound on a moist mineral-matter-free basis.

substitute natural gas (SNG) – a synthetic gas conforming to natural gas standards.

superficial velocity – the linear velocity of a fluid flowing through a bed of solid particles calculated as though the particles were not present.

superheater – a heat exchanger which adds heat to the saturated steam leaving a boiler.

syncrude – synthetic crude oil; oil, produced by the hydrogenation of coal or coal extracts, which is similar to petroleum crude.

synthesis gas – a mixture of hydrogen and carbon monoxide which can be reacted to synthesize a hydrocarbon.

tail gas – a gas issuing from a gas-treatment unit which may be recycled to the process or exhausted.

tar (coal) – a dark brown or black, viscous, combustible liquid formed by the destructive distillation of coal.

therm – a unit of heat used as a basis for the sale of natural gas; equal to 100,000 Btu.

thermal recovery (TR) – a petroleum recovery process that utilizes heat to thin viscous oil in an underground formation and allows it to flow more readily toward wells through which it can be brought to the surface.

topping cycle – the higher temperature thermodynamic power cycle of a combined-cycle system.

turned down – the reduction of reactor flow rates to a fluidized-bed reaction vessel.

ultimate analysis – the analysis of coal based on the percentages of chemical elements.

volatile matter – those constituents of coal, exclusive of moisture, that are liberated from a sample when heated to 1,750°F (for 7 minutes) in the absence of oxygen.

water gas – gas produced by the reaction of carbon and steam to provide mixtures of carbon monoxide and hydrogen; similar to synthesis gas.

water gas shift – the reaction between water vapor and carbon monoxide to produce hydrogen and carbon dioxide or the reverse:  $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{H}_2 + \text{CO}_2$ .

working fluid – a gas stream which directly does work, e.g., powering a gas turbine.

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